University of Alberta

Disposal of Sulphur and Salt in Mined Out Pit at Syncrude

by

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Dedication

To the ones who supported me from the home front:

Nazia, Zuhaib and Shoaib

ABSTRACT

Disposal of sulphur and salt produced as by-products of bitumen upgrading and recycled water treatment is a challenge facing the oil and gas industry. Feasibility of permanently sequestering the salt within solid elemental sulphur was evaluated through an elaborate testing program as well as through use of an analytical model. Tests were performed to observe the flow behavior of molten sulphur as well as salt mixed with molten sulphur.

If the temperature of either the sulphur or outside air increases, the significant flow length increases. The concentration of the deposited salt was enhanced near the discharge outlet and the concentration dispersed at the limits of flow deposit. Segregation of salt near the flow outlet dictates the flow behavior of the combined sulphur and salt mixture. A field disposal option recommends use of a central discharge that would encapsulate the salt within the central core of the block as the molten sulphur solidifies.

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1. INTRODUCTION

1.1 General

The most common source of elemental sulphur in the world is the oil and gas industry. Waste sulphur is currently a challenge for this industry due to the market situation. For quantities of sulphur generated, market requirements will be measured in millions of tonnes per year for the foreseeable future. The current glut in the market has rendered sale of sulphur uneconomical for much of the oil and gas industry (EUB, 2004). Meanwhile, the generation of sulphur from natural gas and bitumen processing to meet environmental regulation is increasing. This leads to a difficult situation with an ever increasing stockpile of excess sulphur requiring proper management. The common practice of storing sulphur blocks in the outdoors without shelter leads to detrimental environmental impact on soil and groundwater.

Molten sulphur production at Suncor Energy Inc. Oil Sands with their Millennium expansion will be about 1200 tonnes per day and at Syncrude Canada Ltd. production is projected to reach about 2700 tonnes per day following UE-1 commissioning.

The extraction of bitumen from the oil sands requires use of large volumes of heated water. This water is either makeup water imported onto the oil sands lease from the Athabasca River or recycled water from the bitumen extraction process. Due to the extensive recycling of water used in the bitumen extraction circuits, dissolved salts are building up in the waters stored on the mine sites.

Syncrude Canada Ltd. has been working to fulfill a full-scale zero liquid discharge (ZLD) system to recover recycle water at the Mildred Lake site in Alberta. The proposed ZLD system uses Electrodialysis Reversal (EDR) to recover approximately 85% of the water for reuse in the extraction circuit. The EDR brine reject is concentrated further in a Brine Concentrator. The highly concentrated brine from the Brine Concentrator is then processed in a forced circulation crystallizer to recover the

remaining water and reduce the brine waste stream to dry solids. The solids produced by the ZLD system will then be suitable for disposal (RCCI, 2001).

The combined large amounts of waste sulphur and salts are increasing every day. Developing a method of disposal for the sulphur and waste salt therefore is very important. The suggested method should be able to overcome all inherent limitations and should be economically viable.

This research is directed towards evaluation of the feasibility of permanently sequestering the salt crystals within the solidified elemental sulphur. The research is directed at studying the various technical aspects of disposing of the molten sulfur mixed with salt through deposition in the mined out pit at the Syncrude mine site.

1.2 Objective of the Thesis

This thesis documents an investigation into developing a procedure for permanent sequestering of salt crystals within solid elemental sulphur. The objective is to permanently separate the salt from coming in contact with surface water through production of and placement of a synthetic sulphur bed rock at the base of a mined out pit. This bed rock will be composed of elemental sulphur that contains a certain percentage of waste salt crystals. The salt crystals will be mixed with the molten sulphur and the mixture will then be deposited at the pit bottom. As the molten mass spreads, it will cool and the molten sulphur will crystallize thus locking the individual salt crystals into this crystallized mass of sulphur.

The main objective of this research is to establish:

- The behavior of molten sulphur after being discharged at different discharge and ambient air temperatures.
- The spreading / flow behavior of molten sulphur that contains various amounts of waste salt

To accomplish these tasks, experimental work was conducted to verify the spreading / flow behavior of sulphur with different salt mix concentrations. The prime focus of this investigation was the development of practical guidelines for the mixing and spreading of sulphur and salt mixture of various concentrations.

The research program was carried out to study the flow behavior of salt and sulphur. The significant length (length that hot sulphur will travel before solidifying) is measured experimentally and then modeled analytically under different sulphur and ambient air placement temperatures. The flow characteristics of the various mixtures of molten sulphur containing salt were investigated. The effect of variation of salt concentration on the flow pattern of the mixture and the length it flows, prior to crystallization, was observed. This research is carried out in collaboration with a companion study presented by Ding (2006) which encompasses the environmental impacts of disposal of salt mixed with sulphur into a mined out pit.

1.3 Scope and Organization of the Thesis

Chapter 2 describes the background, properties of sulphur and salt with major emphasis on heat exchange properties of sulphur, its origin, gap between supply and demand for sulphur and present approaches for its disposal. Origin and properties of salt from the EDR unit are also outlined in this chapter.

Chapter 3 describes the experimental setup to study the spreading behavior of the sulphur salt mixture under various test conditions.

Chapter 4 and 5 outline the experimental results and present a discussion of these results with a focus on the implication of the experimental results on the design of a full scale disposal facility.

Chapter 6 outlines the conclusion of the study and recommendation for additional work.

2. BACKGROUND

2.1 Introduction to Sulphur

Sulphur is one of the few chemical elements that was available to the ancients. It therefore has a long history of both good and evil uses! Free volcanic elemental sulphur was the main source of sulphur until the development of an in-situ hot water mining technique called the Frasch Process in the late 1800's. This rapidly became the source of choice because of the higher purity and in particular the absence of trace contamination of arsenic, tellurium and selenium contamination. Until the boom in the "oil age", over 80% of the world's 25,000,000 tonnes sulphur supply was obtained through this mining technique (Enersul, 2004).

The explosion in oil production and consumption in the second half of the twentieth century saw a complete reversal in the available sources of elemental sulphur. Sulphur recovered from oil and gas processing has risen to 80% of the world's sulphur supply (some 40,000,000 tonnes/year) by the end of the twentieth century. This high quality, involuntarily recovered sulphur has effectively replaced much of the world's mined sulphur in the market place (Enersul, 2004).

The storage, handling, transportation and safety aspects of dealing with elemental sulphur have also undergone drastic change. Mined solid elemental sulphur, was once shipped in wooden barrels via sailing ship, has been replaced by 50,000 tonnes cargoes in marine bulk carriers loaded from 10,000 tonnes unit trains that transport specially formed granules, pellets, pastilles or prills through sophisticated terminals around the world. Safety and environmental concern are now paramount, a far cry from the slave sulphur miners in the volcanic pits of ancient times. Elemental sulphur is now also handled in its liquid form using pipelines and tanks, and by road, rail and ship tankerage.

Sulphur is the preferred raw material for the manufacture of sulphuric acid, one of the world's most important and largest tonnage industrial chemicals. Much has been

made of sulphur's once primary use as an ingredient in explosives however, today's sulphur, as sulphuric acid, is an essential ingredient in the manufacture of phosphate fertilizers (Canadian Minerals Yearbook, 2000). Almost two thirds of the world's annual consumption of elemental sulphur is used to make fertilizers to enhance the world's food supply (Enersul, 2004).

The world's 20 million tonnes of excess sulphur inventory (15 million tonnes of which resides in Canada) is not readily accessible and represents six months of consumption. Storage is in the form of 'blocks,' rectangular structures constructed through the gradual placement of layers of molten sulphur allowed to solidify. The construction of a block involves pouring molten sulphur inside an enclosure and allowing it to solidify. Following solidification, the sides of the enclosure are raised and the process is repeated to a height of more than 10 m. Although (with proper site preparation) this method is environmentally benign and has been historically well accepted, concerns regarding its visual and long term (10 to 40 years) impacts are increasing (Enersul, 2004). Studies are therefore under way regarding various methods for burial, injection into salt caverns and re-injection of hydrogen sulfide underground primarily into reservoirs.

Reclaiming for reuse or sale of sulfur blocks normally takes place with the aid of a variety of melting devices. Thirty years ago excavating equipment simply crushed a block into pieces, which could then be loaded onto trucks, railcars or ships. However, due to the obvious environmental concerns associated with dust emissions during trans-shipment, palletized or granulated sulfur has almost universally replaced 'crushed bulk' for transport of solid sulphur. Sulphur consumed within North America, Europe and Japan is almost entirely transported in its molten state in contrast with shipping of solidified sulphur in other jurisdictions (Enersul, 2004).

In spite of the large volumes traded internationally, commercial transactions are not transparent. The oil industry's traditional 'posting' system is absent in most exporting or consuming regions. Price indications come from reports published in

various fertilizer newsletters. Severe price fluctuations occur as buyers and sellers take advantage of one another's temporary market weakness. This volatility evolved concurrently with the growth of recovered (oil and gas) sulphur and the disappearance of voluntary (Frasch mined) sulphur. A worldwide price collapse took place in 2001 as the USA's phosphate fertilizer industry was suddenly curtailed. Refiners and gas plant operators throughout much of the world had to start paying to have sulphur removed from their installations. Today, while the price has rebounded from zero to about US$40/tonnes in Houston, most operators are experiencing positive feedbacks, but they are still 'giving away BTUs' in an essential chemical element for which there is no substitute (Enersul, 2004).

2.1.1 Sulphur in Alberta

Three sources of sulphur production currently exist in Alberta; namely, processing of sour natural gas, upgrading of bitumen to synthetic crude oil (SCO), and refining of crude oil into refined petroleum products, making Alberta one of the largest sulphur producers in the world (EUB, 2004).

2.1.1.1 Sulphur from Bitumen

Bitumen in oil sands deposits contains significant amounts of sulphur (EUB, 2004). As a result of current upgrading operations in which bitumen is converted to synthetic crude oil, an average of 90 per cent of the sulphur contained in crude bitumen is either recovered in the form of elemental sulphur or remains in other upgrading byproducts including coke.

It is currently estimated that some 208 x 10^6 tonnes of elemental sulphur will be recoverable from the 5.1 billion cubic meters of the remaining established crude bitumen reserves in the surface-mine able area in Northern Alberta. These sulphur reserves were estimated by multiplying the remaining established reserves of crude bitumen by a factor of 40.5 tonnes/1000 m^3 of crude bitumen. This ratio was revised from previous estimates to reflect both current operations and the expected use of high-conversion hydrogen-addition upgrading technologies for the future development of surface-mineable bitumen

reserves. Hydrogen-addition technology yields greater elemental sulphur production than does an alternative carbon-rejection technology, since a larger percentage of the sulphur in the bitumen remains in the upgrading residues, as opposed to being converted to H_2S (EUB, 2004).

2.1.1.2 Sulphur from Bitumen Reserves under Active Development

Only a portion of the surface mineable established bitumen reserves is under active development at the Suncor, Syncrude, and Albian Sands projects. The EUB has estimated the initial established sulphur reserves from these projects to be 67.7×10^6 tonnes. A total of 14.5×10^6 tonnes of elemental sulphur has been produced from these projects, leaving an established reserve of 53.2×10^6 tonnes. During 2003, 1.1×10^6 tonnes of elemental sulphur was produced at the three active projects (EUB, 2004).

2.1.1.3 Sulphur Supply

In 2003, Alberta produced 6.8×10^6 tonnes of sulphur, of which 5.7×10^6 tonnes was derived from sour gas, 1.1×10^6 tonnes from upgrading of bitumen to SCO, and just 20 thousand (10^3) tonnes from oil refining. Sulphur production from these sources is depicted in Figure 2-1. While sulphur production from sour gas is expected to decrease from 5.7×10^6 tonnes in 2003 to 5.1×10^6 tonnes, or some 11 per cent, sulphur recovery from the bitumen upgrading industry is expected to increase to 3.2×10^6 tonnes from 1.1×10^6 tonnes by the end of the forecast period. The Alberta refineries are also expected to replace conventional crude and synthetic crude with bitumen as integration of bitumen upgrading and refining takes place in this forecast period. With this integration, the sulphur recovery will increase from 20×10^3 tonnes in 2003 to 49×10^3 tonnes by 2013. Total sulphur production is expected to reach 8.4×10^6 tonnes by the end of forecast period. Reserves of sulphur as of December 31, 2003 are given in Table 2-1.

2.1.1.4 Sulphur Demand

Demand for sulphur within the province in 2003 was only about 250×10^3 tonnes. It was used in production of phosphate fertilizer and Kraft pulp and in other chemical

operations. Some 97 per cent of the sulphur marketed by Alberta producers was shipped outside the province, primarily to United States, Asia Pacific, and North Africa.

In the early 1990s, a number of traditionally sulphur importing countries installed sulphur recovery equipment on oil refineries and other sulphur-emitting facilities, largely for environmental reasons. Consequently, many of these countries became self-sufficient in sulphur and the price declined significantly (EUB, 2004). Under such low price conditions, many of Alberta's competitors ceased production of sulphur, enabling Alberta's market share to rise throughout the late 1990s. In 2002 and 2003, China increased its sulphur imports from Canada substantially. Increased global demand for sulphur resulted in a major price change, from $16 Cdn /tonnes in 2001 to $40/tonnes in 2003. The export demand for sulphur is expected to increase over the next few years. Demand for Alberta sulphur, both for domestic and export, is expected to rise slowly, reaching 7.5×10^6 tonnes per year by the end of the forecast period. Figure 2-2 depicts the Alberta demand and sulphur removal. This illustrates that stockpiling of some sulphur is required for the foreseeable future.

2.1.1.5 Imbalances between Sulphur Supply and Demand

Because elemental sulphur is fairly easy to store, imbalances between production and sales have traditionally been accommodated through net additions to or removals from sulphur stockpiles. If demand exceeds supply, as was the case over the period 1985-1991, sulphur is withdrawn from stockpiles; if supply exceeds demand, as has been the case since 1992, sulphur is added to stockpiles. Sulphur stockpiles are expected to grow until markets recover from the current world wide glut. Changes to the sulphur inventory are illustrated in Figure 2-2. This indicates the sulphur will be stockpiled through this period as supply exceeds demand.

2.1.2 Disposal of sulphur

2.1.2.1 Relevant Legislation in Alberta

Alberta has a broad frame work of legislation on hazardous waste management. Alberta User Guide for Waste Managers (2001) has been published to assist with this

waste management. Generally, Alberta Environment (AENV) is the government agency responsible for the regulation of hazardous waste in Alberta. For upstream oilfield wastes this regulatory responsibility belongs to the Alberta Energy and Utilities Board (EUB).

There are currently two specific AENV guideline documents related to disposal of waste sulphur (Lau, 2003)

- Guidelines for the Disposal of Sulphur Containing Solid Wastes (AENV, 1983)
- Guidelines for the Remediation and Disposal of Sulphur Contaminated Solid Wastes (AENV, 1996)

Solid sulphur waste generators and handlers are encouraged to reduce, reuse and recover whenever practical. The hierarchy of waste management should be sulphur recovery, in situ land surface treatment and application, followed by land filling, which should be a last resort (AENV, 1996a).

2.1.2.2 Regulation and Guidelines of EUB

Many guides have been prepared by EUB based on legislation in Alberta. The guides most relevant to the management of waste sulphur are:

- Guide 58: Oilfield waste management requirements for the upstream petroleum industry (EUB, 1996)
- Guide 55: Storage requirements for the upstream petroleum Industry (EUB, 2001)

In 1996, the memorandum IL 96-7 clarified that EUB is responsible for ensuring that sulphur by-products are stored so that the future recovery is maximized, while AENV is responsible for ensuring that the storage of these materials does not result in significant negative environmental impacts (EUB, 1996).

2.1.2.3 Implications

The previous presentations outline the abundance with which the sulphur is now produced and due to increasing gap between supply and demand, disposal of ever increasing inventory of sulphur has become a major issue. The above legislations on one side provide clear guidance to prevent environmental degradation and on the other hand make the producers responsible for ensuring that sulphur by-products are available for future recovery. Thus generators of waste sulphur therefore need to consider innovative storage solutions for the longer term in an environmentally acceptable manner with an option for future sulphur recovery.

2.1.3 Conceptual Design Basis for Sulphur Storage Options

To determine a viable storage option (placement) for the surplus sulphur being extracted at the Syncrude site, a detailed literature review was conducted. The following sections briefly outline the findings of this review:

2.1.3.1 Surface Storage Option:

AMEC (2003) were entrusted with the task of providing recommendations for storage of sulphur at various sites in Alberta. The technical review team of AMEC recommended the following two options with respect to storage of sulphur blocks in their interim draft report (AMEC, 2003). Figure 2-3 and 2-4 shows a typical sulphur blocks in Alberta.

2.1.3.1.1 Exclusion Approach

It should be possible to construct a sulphur block using an exclusion approach based on the following assumptions (AMEC, 2003).

- sulphur generation rate of 1,000 tonnes per day (approximately 500 m^3 per day)
- annual precipitation of 450 mm per year
- molten sulphur when poured contains less than 2% moisture content within the pore spaces of the sulphur.

The configuration of a sulphur block according to the above mentioned assumptions will require about 10,000 m^2 (50 m x 200 m) foot print area based on a maximum lift thickness of 5 cm with 500 m^3 of sulphur poured per day. Given that the final surface of block will need to be sloped to allow drainage from the surface, it is believed that the most efficient configuration for the Exclusion Approach block would be to construct berms around the perimeter of the block. The block would then expand in area as the block grows in height up to a height equal to the elevation of the top of the berms. The block would then decrease in size as the upper surface is sloped outward to allow for final capping of the block. Assuming the perimeter berms to be 6 m high with inside slopes of 2:1 and slope above the berm to be 1 horizontal to 4 or 5 vertical, the following areas, volumes and life span of the sulphur block are projected.

- Base area 10,000m^2
- Area at top of berm 16,600 m^2
- Area at top of block 15,000 m^2
- Height of block 12 m
- Sulphur storage volume 175,000 m^3
- Life span of storage area 365 days

2.1.3.1.2 Controlled Approach

The controlled approach is based on handling and storing the sulphur using operational procedures that will significantly reduce the potential for sulphur by-product generation.

The construction of a sulphur block footprint by this approach involves sizing its construction such that the storage area has the capacity to store many years of sulphur production at the facility. This approach typically results in portions of the sulphur block being inactive for lengthy periods while sulphur is being placed to other areas of the block. The controlled approach to sulphur blocking may require a re-evaluation of this approach with the following concerns.

- Water infiltration into the block must be reduced to the minimum practical amounts.
- The sides of the block must be buried with earth as soon as possible after pouring of the sulphur.

In order to achieve these two objectives, a block footprint must be selected such that the surface of the block can receive fresh sulphur on a continuous basis, if desired. Figure 2-5 shows the pouring of molten sulphur on a block at Syncrude. Figure 2-6 and 2-7 shows conceptual cross section and plan of storage facilities used in the industry.

2.1.3.2 Subsurface Storage Option

HAZCO is involved in the conceptual design recommendation for an underground storage facility. The design is based upon disposal of 1500 tonnes per day. The sulphur will be poured onto a bottom liner and contained by aluminum forms. The sulphur will be poured in depths of less than 8 cm and will then be allowed to cool. Cooling takes place quickly as the sulphur adjusts to ambient temperature. The surface area required for this will be around 9400 m^2 (HAZCO, 2002).

2.1.3.3 Relationship between Conceptual Design and This Research Project

The above mentioned approaches recommend the storage of sulphur with respect to the required foot print area. The determination of optimum configuration for a given foot print area depends on the volume and disposal rate of sulphur. The current sulphur disposal practice at Syncrude is also in the form of surface sulphur blocks. The current research is related to the disposal of sulphur and salt in the mined out pit at Syncrude in the form of a below ground block. The research is focused to find the realistic flow behavior of sulphur as well as salt mixed with the sulphur. The flow behavior of the salt and sulphur mixture has been hypothesized as being either homogeneous flow (spreading evenly on the entire footprint) or segregated flow (contained within a specific portion of sulphur block). The significant lengths of sulphur as well as salt sulphur mixture will be measured / calculated with the primary aim of determining its spreading characteristics.

These significant lengths may then be used to assist in finding the optimum design configuration of sulphur salt mixtures when disposed of.

2.1.4 Properties of Sulphur

Sulphur is the fifteenth most common terrestrial element, and the ninth most abundant element in the universe. It is widely distributed in nature in different compounds as well as in pure form; sulphur is often referred to as brimstone. Sulphur is of great industrial importance, though relatively little (ca. 10 %) is used in the elemental form, for the production of carbon disulfide, rubber, pesticides, pharmaceuticals, and cosmetics. By far the greatest amount is used in the form of sulphuric acid and sulphur dioxide.

2.1.4.1 Chemical Properties

Sulphur is a nonmetallic element of group 16, with valence from (-II) to (+VI), atomic number 16, and atomic mass 32.064. It is tasteless and odorless.

2.1.4.2 Physical State

Elemental sulphur occurs in several different allotropic forms, which differ in solubility, relative density and crystalline form, etc. Sulphur can assume many forms depending upon its temperature and impurities, e.g., color, mechanical and thermal properties. Pure elemental sulphur is a bright yellow. The actual color of sulphur often indicates its purity. Bright yellow sulphur is over 99% pure and purity to 99.999% is commercially available.

2.1.4.2.1 Solid Sulphur

Sulphur is known to exist in an unusually large numbers of forms or allotropes. Sulphur exhibits allotropy of two main types; the different ways by which the atoms are combined to form molecules and, the different ways by which the molecules pack to form crystals. Even at room temperature, sulphur can exist in twelve different allotropes. This effect and the loose system of nomenclature have caused much confusion in the past.

Only three physical forms of sulphur are of practical interest here;

- Orthorhombic or rhombic (α) sulphur;
  - The most stable form at room temperature and stable up to about 96 °C
  - It melts at about 113 °C converting completely to monoclinic sulphur
- Monoclinic (β) sulphur;
  - Forms above 96 °C from orthorhombic sulphur, the stable form at melting point
  - It melts at about 119 °C
- Polymeric sulphur (or plastic sulphur)
  - Obtained by quenching hot, viscous liquid sulphur
  - Insoluble in carbon disulphide

Sulphur burns with a pale blue flame, and hence the old English name brimstone, meaning a stone that burns. Sulphur dust may explode with an ignition source and fire hazard is increased if hydrogen sulphide is present (Environment Canada, 1984). Densities and thermal properties, heats of phase transition of sulphur, specific heat, and thermal data for sulphur crystal are discussed later as all are of prime importance in this research. Elemental sulphur is insoluble in water but readily dissolves in liquid ammonia. Orthorhombic and monoclinic sulphur are rapidly soluble in carbon disulphide but not so polymeric sulphur (Meyer, 1965).

There is substantial reactivity between sulphur and various solvents. At room temperature, sulphur reacts with several inorganic solvents and some amines and nucleophilic reagents but not with hydrocarbons or aromatic compounds. Above 150 °C it reacts with every known inorganic and organic solvent. Sulphur is also known to react violently in contact with oxidizing agents or powdered metals (Environment Canada, 1984). Heat, light or catalysts can initiate reactions of sulphur with organic compounds. It has been observed that the solubility of orthorhombic sulphur in organic solvents reduces and the precipitate re-dissolves very slowly after sulphur is exposed to sunlight.

2.1.4.2.2 Liquid Sulphur

There is a wide range of melting points for sulphur because different forms of sulphur usually co-exist. The melting and solidification temperature points are determined by the history of sulphur, i.e., the relative concentration of various metastable species which can act as solute in S^{β} and cause a solidification temperature point depression. Melting points of some common allotropes of sulphur are given in Table 2-2.

For pure orthorhombic sulphur, it turns a pale yellow, transparent liquid of low viscosity at about 113 °C. The behavior of the sulphur shows no unusual changes up to about 159 °C, at which point, its viscosity increases abruptly. The high viscosity is caused by long strands of sulphur molecules being entangled with each other and thus resisting the smooth flow of the liquid. The 159 °C temperature is the polymerization temperature as well as the point of minimum density for liquid sulphur (Meyer, 1977).

2.1.5 Properties Relevant To Heat Exchange and Flow

2.1.5.1 Viscosity

Viscosity is the resistance of a fluid to flow. This resistance acts against the motion of any solid object through the fluid and also against motion of the fluid itself past stationary obstacles. Viscosity also acts internally on the fluid between slower and faster moving adjacent layers.

All fluids, i.e., all liquids and gases, exhibit viscosity to some degree. Viscosity may be thought of as fluid friction, just as the friction between two solids resists the motion of one over the other but also makes possible the acceleration of one relative to the other (e.g., the friction between the wheels of an automobile and a highway), so viscosity resists the motion of a solid through a fluid but also makes it possible for a propeller or other device to accelerate the solid through the fluid.

The kinematic viscosity is the ratio of the viscosity to the density. With viscosity measured in poises and the density of grams per cubic centimeter, the unit of kinematic

viscosity is stoke, with the units square centimeters per second. In the SI system of units, viscosities are expressed in N-s/m^2 (or Pa.s) and kinematic viscosities in either m^2/s or cm^2/s.

As shown in Figure 2-8, the maximum viscosity of pure liquid sulphur occurs at 196 °C, followed by a gradual decrease with higher temperatures. These viscosity changes are noted to be perfectly reversible. At higher temperatures, the mole fraction of the polymer increases. This is accompanied by development of shorter, reactive molecular chains which account for the incredible high reactively of hot liquid sulphur towards traces of organic compounds. This characteristic enables the detection of impurities in sulphur reliable through the display of darker color from reaction products (Meyer, 1977).

Liquid viscosity data are important in many engineering applications in the chemical processing and petroleum refining industries. The correlation for liquid viscosity as a function of temperature is given by the equation shown below (Carl, 1995).

$$Log_{10}N_{liq} = A + B/T + CT + DT^2 \qquad [2\text{-}1]$$

where;

N_{liq} = viscosity of liquid, centipoises

A,B,C and D = regression coefficients for chemical compound

T = temperature, K

For sulphur;

A= 326.3558

B = -5.5393E+04

C = -6.0794E-04

D = 3.7191E-04

Figure 2-9 shows the viscosity of sulphur for the lower temperature range (Fanelli and Bacon, 1945).

2.1.5.2 Density

Density is the ratio of the mass of a substance to its volume, expressed, for example, in units of grams per cubic centimeter or Mg/m^3. The density of a pure substance varies little from sample to sample and is often considered a characteristic property of the substance.

Liquid density data are important in process engineering design, such as sizing of storage vessels that contain the basic raw materials and products for a plant. Usage is encountered in various heat, mass, and momentum transfer operations.

The modified form of the Rackett equation was selected for correlation of saturated liquid density as a function of temperature (Carl, 1995)

$$\text{Density} = A\, B^{-(1-T/Tc)n} \qquad [2\text{-}2]$$

where;

Density = saturated liquid density, g/ml (Mg/m^3)

A,B, and n = regression coefficients for chemical compound

T = temperature, K

T_c = critical temperature, K

For sulphur;

A = 0.2448

B = 0.10440

n = 0.11400

T_c = 364.00

Table 2-3 outlines some precise density measurements of highly purified liquid sulphur reported by Matson (1954).

2.1.5.3 Thermal Properties

Thermal conductivity is defined as the amount of heat per unit time per unit area that can be conducted through a plate of unit thickness of a given material, the faces of the plate differing by one unit of temperature. Thermal conductivity is a property of

materials that expresses the heat flux (f) (W/m^2) that will flow through the material if a certain temperature gradient ΔT (K/m) exists over the material.

The thermal conductivity (K) is usually expressed in W/m.K. The usual formula is:

$$f = K * \Delta T \qquad [2\text{-}3]$$

It should be noted that thermal conductivity is a property that describes the semi static situation; the temperature gradient is assumed to be constant. As soon as the temperature starts changing, other parameters enter the heat flow equation.

In case of changing temperature conditions, the **heat capacity** C ($J/K.m^3$) plays a role. The heat capacity is again a material property. It expresses the fact that for changing the temperature ΔT of a certain volume V (m^3) of material an amount of energy must flow in or out of the material. The heat capacity is usually linked to the density ρ (kg/m^3) of the material. The heat capacity is usually expressed in the textbooks as specific heat capacity (Cp) (J/K.kg), which must be multiplied by the density to get the volumetric heat capacity.

$$C = \rho * Cp \qquad [2\text{-}4]$$

When dynamic processes are involved, the change of temperature versus time, at known boundary conditions is determined using both thermal conductivity and heat capacity. The thermal diffusivity α (m^2/s) is expressed as:

$$\alpha = K / [\rho * Cp] = K/C \qquad [2\text{-}5]$$

One approach used to find the thermal conductivity of sulphur is K in W/(m K) (Carl, 1995).

$$K = A + B\,T + C\,T^2 \qquad [2\text{-}6]$$

where;

A = 0.0262

B = 4.1738E-04

C = -3.2471E-07

Enthalpy is the measure of the heat content of a chemical or physical system; it is a quantity derived from the heat and work relations studied in thermodynamics. Since the enthalpy of a fluid is a function of its temperature and pressure, the temperature dependence of the enthalpy can be found by measuring the rise in temperature caused by the flow of heat at constant pressure. The constant-pressure heat capacity C_p is a measure of the change in enthalpy at a particular temperature. Similarly, the internal energy is a function of temperature and specific volume. The constant-volume heat capacity C_v is a measure of the change in internal energy at a particular temperature and constant volume.

Because the specific volumes of solids and liquids are generally smaller, then unless the pressure is extremely high, the work done by an applied pressure can be neglected. Therefore if the enthalpy can be represented by the internal energy component alone, the constant-volume and constant-pressure heat capacities can be said to be equal.

Therefore for solids and liquids: $C_p = C_v$

The **specific heat capacity** represents the amount of energy required to raise 1 kg by 1 °C and can be thought of as the ability of a substance to absorb heat. Therefore the SI units of specific heat capacity are J/Kg-°K (J/Kg-°C). Water has a very large specific heat capacity (4200 J/Kg-°K) compared with many fluids. The specific heat capacity of sulphur (liquid) is 1006 J/Kg-°K [Lewis and Randall (1911) and Touloukin (1970)].

2.1.6 Comparison of Properties of Water and Sulphur (Liquid)

In studying the flow properties of sulphur, it is interesting to compare its properties with water. The working range of temperature in the case of sulphur is between 115 °C to 140 °C and in the case of water is 0 °C to 10 °C. In this temperature range both these materials have almost the same mobility behavior. This is the range in

which sulphur has its least viscosity and is the range of interest for the project. The density of sulphur is around 1800 kg / m^3 whereas the density of water is 1000 kg / m^3. The absolute viscosity of sulphur is about ten times greater than that of water. The specific heat of water is around four times higher than that of sulphur. For this temperature range, the comparison is shown in Table 2-4.

2.2 Salt

The word salt is used for the contaminants which will be separated from recycle water during treatment. To obtain salt for use in this research program brine from the treated water received from Syncrude was dried in an oven and then it was used in its solid form. This dried mass of solid is referred as salt throughout the thesis.

2.2.1 Properties of Salt

2.2.1.1 Chemical Analysis

Chemical analysis of this salt is summarized in Table 2-5 (Ding, 2006). The samples were sent to four different laboratories and the following outlines the sources of each analysis.

- Source 1: Test Report on Salt Cake, by Lafarge Canada, Inc.
- Source 2: RCCI test report — Bench-scale Crystallizer Testing, Concentration and Crystallization of EDR and Evaporator Pre-concentrated Recycle Water.
- Source 3: Analytical Report on Dissolved Salt Solution, by Norwest Labs.
- Source 4: Compositional Analysis of One Sample of Brown Solids Identification as 319, by AGAT Laboratories, Calgary.

Note: The University of Alberta produced the samples for Sources 3 and 4 and the other were provided by Syncrude Canada Ltd directly for the separate individual analysis.

2.2.1.2 Specific Gravity

The measured specific gravity is 2.63 as reported by Ding (2006).

2.2.1.3 Particle Size Distribution

The salt was obtained by drying the brine solution in an oven at 105 °C. The solid obtained from the drying pans was not of any specific size or shape. They were in small chunks and platelets. These chunks were then crushed into small pieces for use in the research program. Figure 2-10 shows the gradation curve of solids (salt) used in the research and Figure 2-11 represents the gradation curve of the commercially available salt used in the separate flow experiments.

2.3 Salt and Sulphur Mixture

Salt and sulphur were mixed to create slurry in the temperature range 120 to 140 °C. This slurry was anticipated to act as either a segregating or non-segregating slurry. The phenomenon of segregating or non-segregating slurry has been described by Küpper (1992) and is briefly discussed in the following sections to understand how the mixture behaves when discharged.

2.3.1 Segregating and Non Segregating Slurries

Segregation of slurry refers to the tendency of the solid fraction (or part of it) to settle from the carrier fluid, creating a concentration gradient within the mass. Other terminologies found in the literature to describe this difference in behavior include: settling/non-settling, heterogeneous / homogenous, bleeding / non-bleeding and stream flow / mudflow (Blissenbach, 1954; Hooke, 1967, 1968; Bull, 1968, 1972; Beaty, 1970; Lustig, 1974; Macke, 1977; Weave, 1984; Pierson and Scott, 1985 among many others). Whether slurry behaves as segregating or non-segregating mixtures depends on the:

- Type of carrier fluid
- Type of solids
- Grain size distribution of solid
- Proportion of solids to the total amount of slurry (slurry concentration)

2.3.2 Non Segregating Slurries

When a slurry behaves in a non-segregating manner, the solids remain uniformly distributed throughout the mass. This non-segregating behavior is common in slurries of high solids concentration and especially with materials containing fine particle sizes. Usually there is a sharp increase in viscosity within the slurry compared to the viscosity of the carrier fluid and the slurry displays non-Newtonian flow behavior. Viscous forces within the slurry dominate and control the flow behavior of the slurry.

2.3.3 Segregating Slurries

When segregating behavior dominates, the solids are generally not evenly distributed and pronounced solids concentration gradients exist within the mass. The fluid and the solid phases interact, but retain separate identities, and the slurry viscosity remains similar to that of the original carrier fluid. In this case, particle inertial effects dominate and influence the fluid flow and depositional behavior.

2.3.4 Properties of Slurry

For a particular type of solids and fluid under constant flow conditions, the properties of the slurry depend on the relative amounts of solids and fluid, i.e., the slurry concentration. Within the solid fraction a distinction must be made between the finer and coarser fraction. The finer fraction of the solids controls many properties of the slurry and may have an important effect on the segregating or non segregating behavior. In some cases, a relatively small change in the percentage of the fines content may change the slurry behavior from one type to the other. Therefore, it is convenient to consider the finer fraction of the solids separately.

Also, segregating/non-segregating behavior depends on the flow conditions themselves. Slurries flowing at a lower energy level (such as in flume tests) display a different behavior compared with the same slurry flowing at higher energy levels (such as under field conditions).

Segregation also has a direct effect on the grain size distribution within the deposit. Flow of segregating slurries results in hydraulic sorting of particles, with different particle size fractions being deposited at different locations along the flow path. Deposits formed by non-segregating slurries do not develop hydraulic sorting, therefore the coefficient of uniformity is relatively high and the grain size distribution remains approximately constant within the deposit and at different locations from the discharge outlet.

2.3.5 Hydraulic Sorting

A sediment laden flow with segregating behavior tends to deposit grains of different sizes in different locations along the discharge path. In general, the sediment transport capacity of the flow decreases progressively and, as a result, larger grains tend to be deposited first and smaller grains are deposited further downstream. Consequently, the typical grain size distribution along a hydraulic fill is characterized by a decrease in mean grain size and an increase in the amount of fines with distance from the discharge point.

This separation of hydraulically deposited materials into different granulometric fractions due to the hydraulic characteristics of the flow is called hydraulic sorting. This phenomenon has also been called hydraulic segregation (Morgenstern and Küpper, 1988; Fourie, 1988; Lighthall et al., 1989 and Conlin, 1989).

2.4 Proposed Research

This research is focused on evaluating the feasibility of permanently sequestering salt crystals within solid elemental sulphur. The salt crystals will be mixed with the molten sulphur and then the mixture will be discharged and allowed to flow on the bottom of the mined out pit. As the molten mass spreads, it cools and the molten sulphur crystallizes thus encapsulating the individual salt crystals into this solidified mass of sulphur.

The flow characteristics of the various mixtures of molten sulphur containing salt crystals will be investigated. Salt concentrations within the molten sulphur will be varied, and the flow pattern of this mass and the travel length prior to its crystallizing will be measured.

2.4.1 Experimental Program

An experimental program was set up to test the physical flow behavior of sulphur with or without salt. The objective of this experimental program was to determine the length of flow (significant length) before the sulphur will solidify after being discharged at a particular flow rate.

An apparatus was fabricated which contained the sulphur at the required temperature and allowed uniform mixing of salt during the experiment. The experiment was conducted with different salt sulphur composition. After the mixture had solidified, samples were collected at different locations and the percentage of salt within the solidified mass was determined. The experiments were used to establish the flow patterns of sulphur and salt under different test concentrations.

The results from the experiment program were compared to the numerical model predictions to assess the flow pattern under different values of mass rate of flow, air temperature, initial temperature and flow for different salt concentrations was studied.

2.4.2 Methodology

The experimental program was used to examine how the mass flowed, the physical attributes of the mass and to evaluate the heat transfer numerical model reported by Otto (2002).

2.4.2.1 Model

The numerical model developed by Otto (2002) was based on freeze separation of dissolved salts from water. In the numerical model, groundwater entering a pond contains specific heat energy based on its initial temperature, the kinetic energy of flow

and any gain from radiant solar energy. As soon as the groundwater enters the pond, it begins to dissipate (lose) heat energy through:

- Conduction
- Convection
- Evaporation
- Internal and external friction
- Radiation
- Heat absorbed in melting snow falling on the flowing liquid
- Potential energy as it flows from the entry point to the lowest point in the pond

In the numerical model used, the only significant energy source was the initial heat energy (i.e., specific heat) and energy released as the material undergoes phase change (latent heat of fusion).

The conceptual model is treated as two separate heat exchange regimes. In the first heat regime, flow occurs in a thin film with constant velocity, mass and heat energy is lost from the mass to the upper and lower surface through conduction, and via convection to the air above. This regime ends when the material decreases in temperature to its phase transition temperature. The average of the initial temperature and the air temperature is used to calculate the heat flow rate in this regime. The total amount of energy, and the flow distance in this regime is a function of the discharge temperature. The second heat exchange regime is based on the rate of latent heat of fusion released. The total amount of energy, and the length required for the second regime is a function of the air temperature, and fluid phase change temperature. The significant length is the sum of the lengths required for heat loss via the two regimes (Otto, 2002).

The energy balance model for the heat exchange in regime 1 per unit width is

$q_{specific} = q_{conduction} + q_{convection}$

where;

$q_{specific}$ = rate of specific energy loss (energy of enthalpy) (J/s m),

$q_{conduction}$ = rate of energy loss via conduction (J/s m), and

$q_{convection}$ = rate of energy loss via convection (J/s m)

The rate of specific heat added to the sulphur is calculated as (Kinsky, 1977)

$q_{specific} = M \Delta H$

where;

$q_{specific}$ = rate of specific energy gain (energy of enthalpy) (J/ s m),

M = mass flow of sulphur per unit width (Kg/s m), and

ΔH = enthalpy (J/Kg)

Conductive heat flow rate per unit width from the sulphur is calculated as (Kinsky, 1977)

$$q_{conductive} = \frac{k_t (T_{avg} - T_{air}) l_{sl}}{b}$$

where;

$q_{conduction}$ = rate of energy loss via conduction (J/s m),

k_t = thermal conductivity (J/[m s K]),

T_{avg} = average sulphur temperature in heat regime 1 (K),

T_{air} = air temperature, assumed equal to design temperature (K),

ls_l = length in heat regime 1 (m), and

b = thickness of boundary layer (m). Various references (Perry and Chilton, 1973; Kay and Nedderman, 1974; Drakos, 1994) give equation to find this value of b.

Convective heat flow rate per unit width from the sulphur is calculated as (Kinsky, 1973)

$$q_{convective} = (T_{avg} - T_{air})\, ls_1\, h$$

where

$q_{convective}$ = rate of energy loss via convection (J/s m),

T_{avg} = average sulphur temperature in heat regime 1 (K),

T_{air} = air temperature (K),

ls_1 = length in heat regime 1 (m), and

h = convective heat transfer coefficient per unit width (J/[s m^2 K])

Using these preliminary equations, a spreadsheet numerical model was used to calculate the total length required for the sulphur to change phase using different discharge temperatures of sulphur with different concentrations of salt.

2.5 Summary

This chapter describes the difference between the supply and demand of sulphur in western Canada. As the gap between supply and demand is increasing, the need for disposal of this sulphur according to regulatory requirements is becoming more extensive. Present day mode of disposal of sulphur at Syncrude plant is discussed. Water which is an important part of the bitumen hydro-transport and upgrading process is becoming more and more contaminated with dissolved salts via the extensive water recycling. The contaminants that will be separated from this recycled water needs to be disposed of. This thesis is about studying the physical aspects of disposing of these contaminants (salt) along with sulphur in the mined out pit at Syncrude.

Properties of sulphur and salt are important in studying the flow behavior of fluid mass containing both sulphur and salt. Viscosity, density, hydraulic conductivity and heat capacity are the major properties governing the flow of sulphur which acts as the carrier fluid for the salt. All these properties vary with the temperature. Sulphur's

melting point is around 115 °C (varying upon type of allotrope). In most studies of liquid sulphur, some striking changes in its physical properties are observed at about 160 °C. The flow properties will be studied within this range of temperature. The contaminants removed from the recycled water were also analyzed and its properties are also discussed.

After discussing the problem facing the industry, a proposal for dealing with the problem in the future was presented. This proposal consists of finding a feasible way of permanently sequestering the salt crystals within solid elemental sulphur. To study this concept, an experimental program was performed. The data obtained will later be compared to the heat exchange model under different mass rate of flow and temperature conditions. This will assist in understanding how best to design a field scale discharge system.

Reserves of Sulphur (10^6 tons)			
	2003	2004	Change
Initial established reserves			
Natural gas	244.7	238.7	+6.0
Crude bitumen[a]	67.7	67.7	0.0
Total	312.4	306.4	+6.0
Cumulative net production			
Natural gas	212.8	207.1	+5.7
Crude bitumen[b]	14.5	13.4	+1.1
Total	227.3	220.5	+6.8
Remaining established reserves			
Natural gas	31.9	31.6	+0.3
Crude bitumen[a]	53.2	54.3	-1.1
Total	85.1	85.9	-0.8

Table 2-1 Reserves of sulphur as of December 31, 2003 (EUB, 2002)

[a] Reserves of elemental sulphur under active development at Suncor, Syncrude, and Albian Sands operations as of December 31, 2003. Reserves from the entire surface mineable area are larger.
[b] Production from surface mineable area only.

Melting Points of Sulphur		
Allotropes	**Melting Point,°C**	**Remarks**
Alpha-S	112.8	Single crystal
	115.1	Microcrystal(more common)
Beta-S	114.5	'Natural"
	119.6	'Ideal' and observed (thermodynamics melting pt)
	120.4	Microcrystal
	133	'Ideal" calculated
Insoluble-S	77; 90; 160	Optical, TDA, DTA
	104	
S_n	75	Optical
	104	Classic

Table 2-2 Melting points of sulphur

Density of Sulphur	
Temp	**Density**
°C	**g/cm^3**
121	1.8037
124	1.8007
127	1.7981
129	1.7957
132	1.7935
135	1.7912
138	1.7888
141	1.7864
143	1.7842
146	1.7818
149	1.7795
152	1.7773
154	1.7752

Table 2-3 Density of liquid sulphur (after Matson)

Comparison of Water and Sulphur							
Water				Sulphur			
Temp	Density	Specific Heat	Absolute Viscosity	Temp	Density	Specific Heat	Absolute Viscosity
°C	(kg/m^3)	(J/ [kg K])	Centi Poise (cp)	°C	(kg/m^3)	(J/ [kg K])	Centi Poise (cp)
0	999.8	4,218	1.79	115	1794.5	997.65	11.91
1	999.9	4,214	1.73	120	1792.1	1003.60	11.15
2	999.9	4,211	1.67	125	1789.7	1010.92	11.01
3	999.9	4,208	1.62	130	1787.3	1018.53	9.97
4	1000.0	4,205	1.57	135	1784.9	1028.43	8.93
5	999.9	4,202	1.52	140	1782.4	1039.85	8.04
6	999.9	4,200	1.47	145	1780.0	1055.07	7.44
7	999.8	4,198	1.43	150	1777.5	1086.89	6.70

Table 2-4 Comparison of water and salt properties at corresponding liquid temperature

Solid Salt Composition				
Mass fraction of ions %	Source 1	Source 2	Source 3	Source 4
SO_4^{-2}	19.1	19.0	20.6	3.5
Cl^-	42.8	44.0	43.0	51.1
CO_3^{-2}				6.5
Na^+	36.7	36.5	34.6	33.8
K^+	0.5		0.7	1.1
Mg^{+2}	0.5	0.44	0.4	0.3
Ca^{+2}	0.4	0.33	0.9	3.9

Table 2-5 Chemical analysis of solid salt composition from different sources (Ding, 2006)

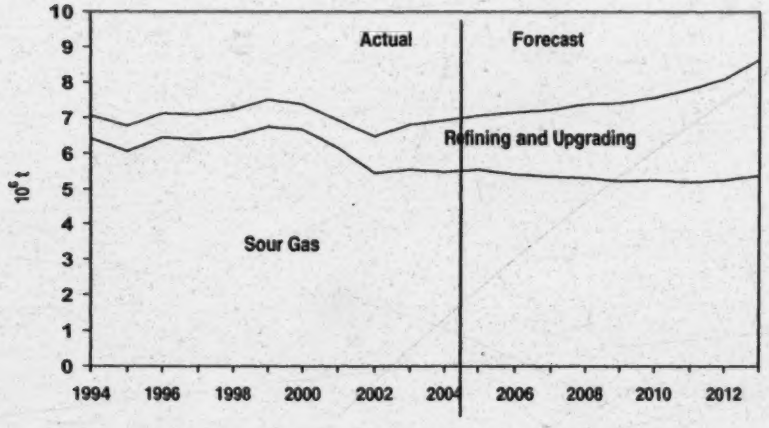

Figure 2-1 Sources of Alberta sulphur production (EUB, 2004)

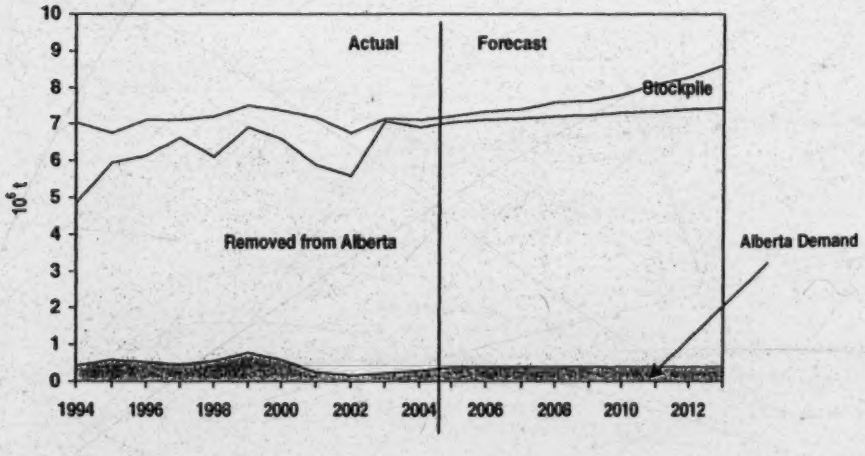

Figure 2-2 Sulphur demand and supply in Alberta (EUB, 2004)

Figure 2-3 Sulphur blocks at Syncrude

Figure 2-4 A typical sulphur storage block (Nagendran, 1978)

Figure 2-5 Sulphur disposal in progress at Syncrude

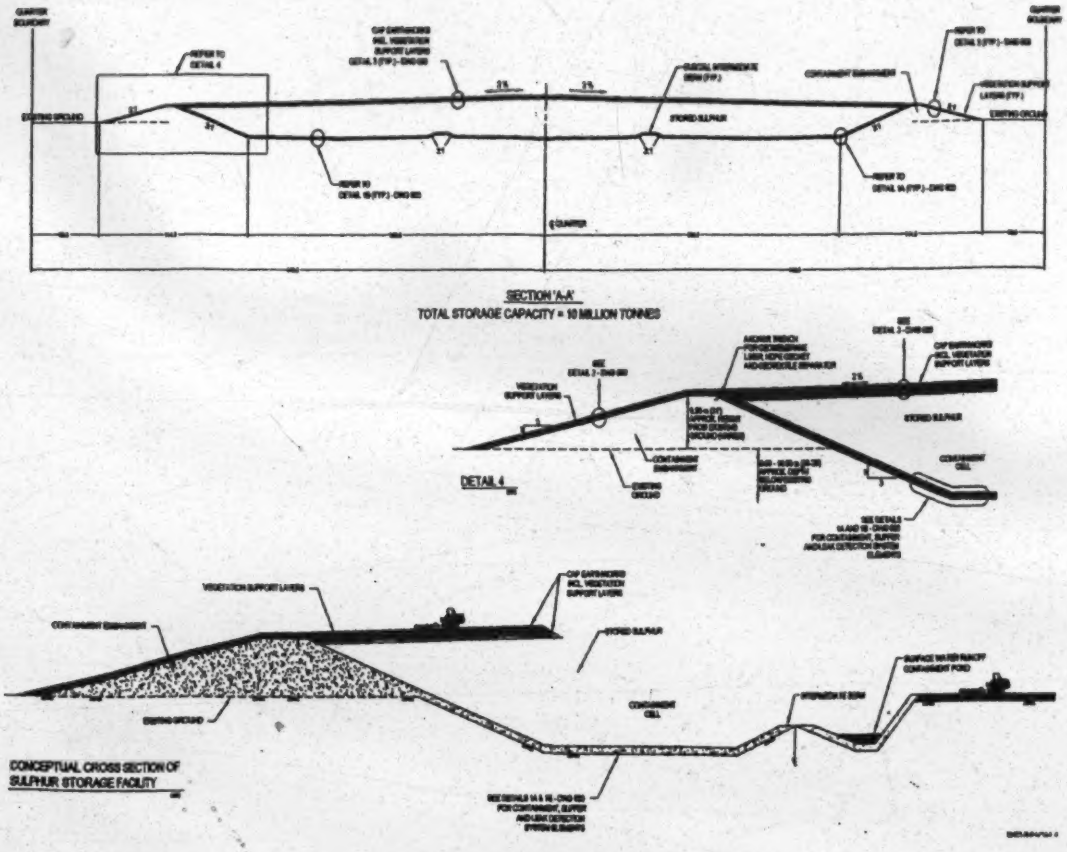

Figure 2-6 Conceptual cross section of sulphur storage facility used in industry (after HAZCO, 2002)

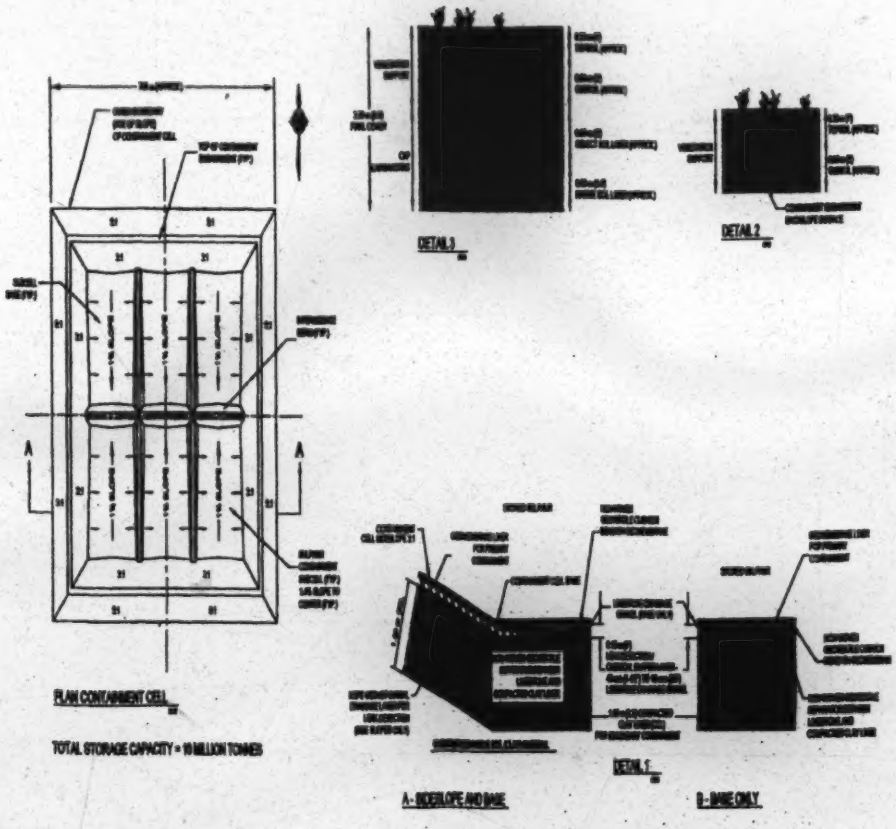

Figure 2-7 Conceptual plan of underground storage facility used in industry (after HAZCO, 2002)

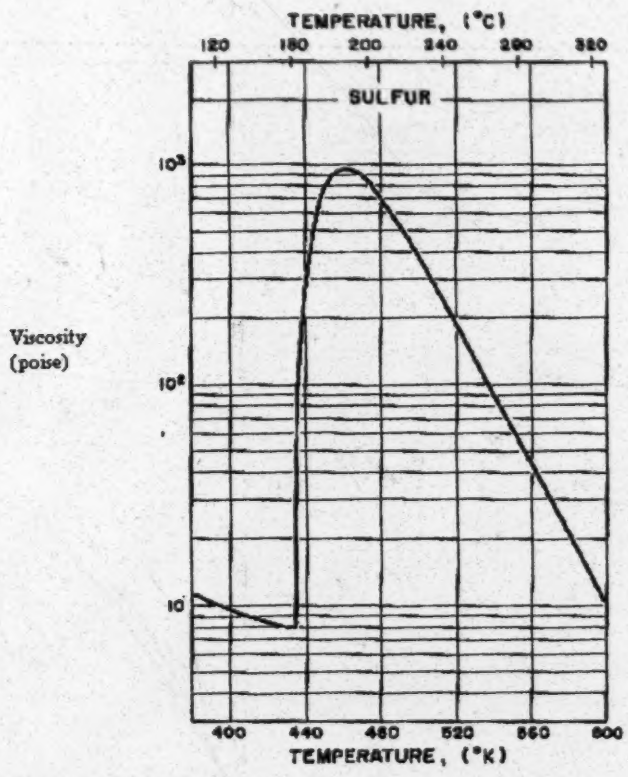

Figure 2-8 Viscosity versus temperature of liquid sulphur (after Mayer, 1965)

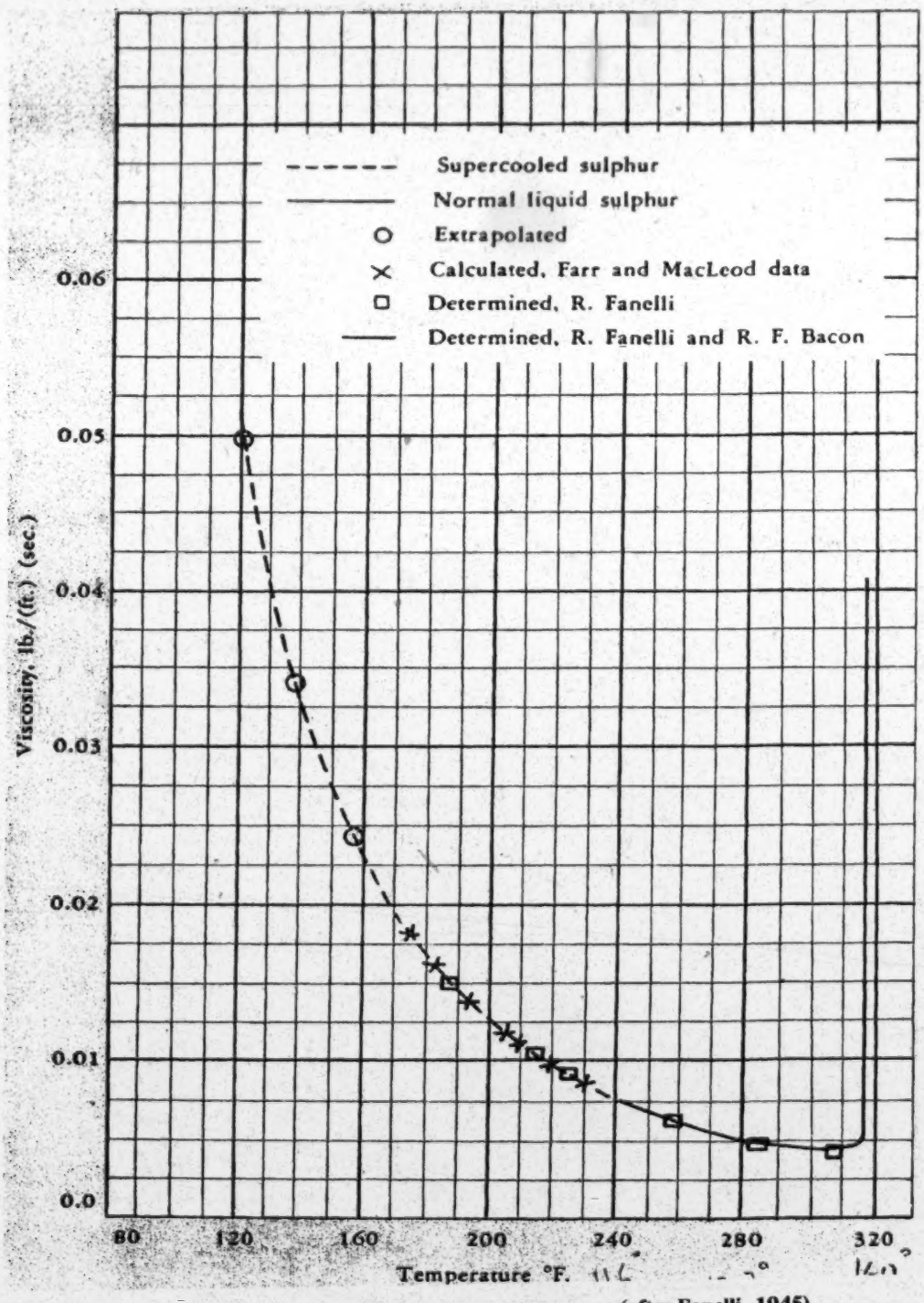

Figure 2-9 Viscosity of liquid sulphur at low temperature range (after Fanelli, 1945)

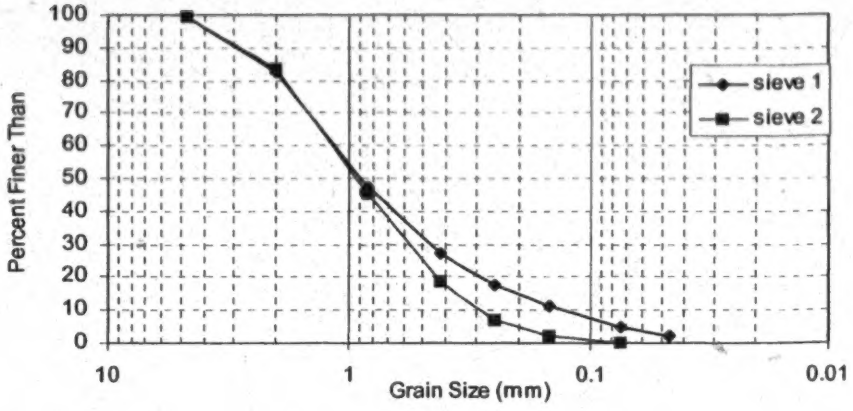

Figure 2-10 Gradation curve of salt solid obtained from oven dried brine

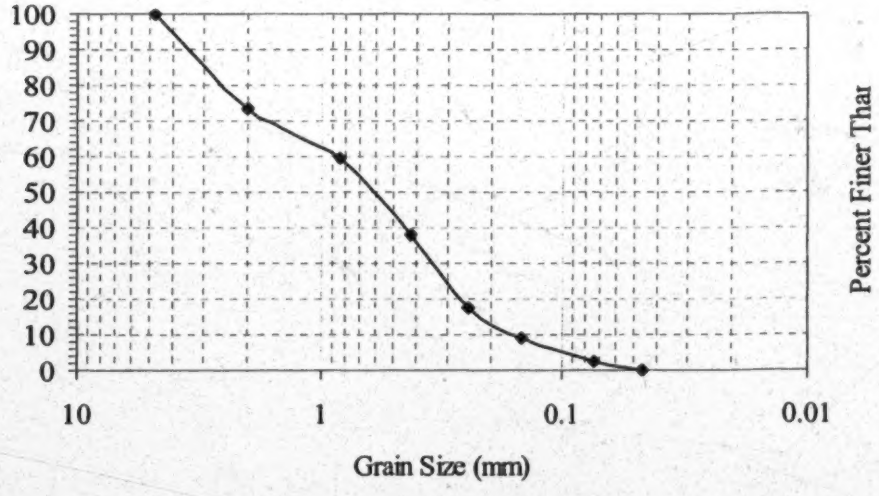

Figure 2-11 Gradation curve of commercially available salt used in research

3 EXPERIMENTAL SETUP

3.1 Introduction

Heat exchange models have successfully been used to analyze freeze separation of dissolved salts from ground water (Otto, 2002). These models are based on loss of heat to the atmosphere through convection and to the ground through conduction. Although the research regarding freeze separation was mainly related to water, the same concepts are applied to the flow of sulphur with and without salt.

In our study not only was the flow of sulphur investigated but the main objective was the behavior of sulphur when combined with different mass concentrations of salt. With the addition of salt in the sulphur, the mixture may behave as either a segregating or non segregating slurry. The physical flow of sulphur and salt will depend upon the temperature, viscosity, density, specific heat, thermal conductivity of carrier fluid and also on the grain size distribution and density of the salt. In order to study the physical flow behavior of sulphur and salt, experiments were performed because no previous work on this topic was found in the literature. Flow of sulphur occurs over a narrow range of temperature in which the flow properties of sulphur will assist with the spreading of sulphur containing salt. The test apparatus was manufactured to assess the flow of sulphur under constant controlled temperature conditions. The other challenges associated with the experimental equipment were to ensure constant mixing of sulphur and salt so that it was discharged with the uniform salt concentration and to keep the discharge volume constant so that mass rate of flow could be maintained.

3.2 Test Site and Experimental Setup

The test was conducted outside the Civil Engineering Building to ensure safety. As the test involved heating of sulphur, there was a possibility of H_2S gas being released. H_2S (Hydrogen Sulphide) gas is produced when molten sulphur comes in contact with water. H_2S is very dangerous gas which can be fatal at high concentrations.

When the pipes were heated to clear the path for molten sulphur, sulphur accumulated in the pipes from the previous tests becomes overheated and flowed out of the pipes releasing pungent odor. These safety concerns required that the experiment be performed in the open air but near by so that safety could be maintained. Figure 3-1 shows the general equipment layout behind the Civil Engineering building.

The test consists of discharging molten sulphur under controlled conditions with and without salt addition. The tests using sulphur alone were an important guide for the subsequent tests of sulphur mixed with salt. The objectives were

- To compare the theoretically predicted flow patterns with those observed during the experiment.
- To test the analytical framework for the test. This includes prediction of the significant length.
- To evaluate the performance of the test apparatus to evaluate its use for tests with salt mixed in molten sulphur.

3.3 Test Equipment

All the tests consisted of discharging sulphur and salt from the apparatus onto a platform to study the flow behavior of each mixture. The equipment was manufactured to keep the sulphur hot while mixing and to ensure the mixture flowed at a constant mass rate. The major components of the equipment are:

1. oil bath,
2. heating system,
3. sulphur vessel,
4. mixer,
5. air line,
6. loading crane, and
7. platform

3.3.1 Oil Bath

The oil bath was a steel cylinder 0.75 m in diameter and 1.33 m in height. Its function was to keep the temperature constant in the sulphur vessel (Fig 3-2) and was generally operated between 115 °C and 160 °C. If the sulphur temperature falls below 115 °C the sulphur will solidify and if it rises above 160 °C the sulphur becomes too viscous to flow due to physical changes in the sulphur.

The oil bath also provided support for the sulphur vessel and for the equipment hanging from the crane. The crane was required to monitor the mass rate of flow by using a load cell attached to the crane to measure the change in the mass during discharge of sulphur mixture (Figure 3-1). The pipe fittings from the sulphur vessel to the outside spigot passed through the oil bath to maintain it at the appropriate test temperature. Figure 3-2 shows the oil bath and sulphur vessel.

The selection of liquid for the oil bath was very important. The fluid selected should have its boiling point higher than 160 °C and it should be safe to handle. Another consideration in selecting the liquid was that it should be capable of retaining heat for an extended period. As the experiments were conducted outdoors in the fall when the air temperatures may fall below zero, the liquid also should not freeze. The quantity of liquid required was around 282 liters. The liquid finally selected was THERMIA* C supplied by Shell. It is a chemically stable heat transfer oil with MSDS No. 407 – 640. Its boiling point was greater then 315°C (Shell Canada Limited, 2001) and it increases in viscosity as it cools to -20 °C.

The oil bath was also wrapped with insulation to retain heat. Fiberglass insulation sheets were wrapped around the oil bath and held in place with steel wire. Four hooks were welded to the oil bath at the top to attach the chains to hang the apparatus from the load cell attached to the crane. The oil bath rested on the steel supports, which in turn rested on the ground. All the pipefitting responsible to allow sulphur flow out of the sulphur vessel was attached to oil bath. The temperature in the oil bath was monitored

with a thermistor attached to a data logger. Heat was provided to the bottom of the oil filled vessel by a propane torch.

3.3.2 Heating System

Heating was required to heat the oil bath to keep the sulphur vessel at its test temperature and to melt the sulphur between tests, and was provided using the two propane torches.

Normally, pre-heated molten sulphur was poured into the sulphur cylinder. The sulphur was supplied by Syncrude in 20 L pails. The solid sulphur was removed from the pails and broken into pieces. It was then heated to melt it and this was poured into the sulphur vessel. The heating of sulphur to test temperature was achieved in two ways

- Overnight it was preheated in a ventilated oven in the laboratory, or
- Preheated in metal pails at the test site using a tiger torch

The oil bath was normally heated 4 to 6 hours before a test to bring the sulphur to the required temperature. The temperature inside the sulphur vessel and in the oil bath was monitored and if the temperature began to rise, the torch was withdrawn to maintain the temperature in the proper range.

The discharge piping also needed to be heated just before the test. The discharge piping which normally contained solidified sulphur from the previous test was heated to melt this sulphur. Then sulphur was discharged from the vessel to clear the pipes. The discharge was collected in a separate metal pail prior to the discharge pipe being directed to the test area.

3.3.3 Sulphur Vessel

The sulphur vessel was placed in the oil bath. Its major functions were:

- To store the sulphur and salt mixture
- To keep the sulphur and salt mixture at the test temperature
- To facilitate mixing of salt within the sulphur
- To keep the mixture under a constant pressure so that a constant mass flow rate during each test could be maintained.

The sulphur vessel was covered with a steel plate and sealed at the top with a gasket. The steel plate acted as support for the motor. A 50 mm diameter hole was placed in the steel plate through which salt was added to the sulphur using a steel funnel. After salt was added to the molten sulphur, the hole was closed with a cap which was fitted with a valve. An air line was attached to pressurize the vessel for control during discharge of the sulphur-salt mixture.

3.3.4 Mixer

The mixer keeps the salt thoroughly mixed within the sulphur during each test so that a uniform flow of the salt sulphur mixture was maintained during the experiment. As the specific gravity of the salt was higher than that of sulphur at the test temperature, the salt settled as soon as it was added to the sulphur. If the salt settled, a uniform mixture could not be discharged. The mixer insured a uniform mixture. The lower end of the mixer shaft rested on a ball at the bottom of sulphur vessel and the top end was attached to the electric motor for rotation. The shaft rod assembly was supported by a collar at the top of sulphur vessel to keep it vertical.

The mixer consists of a 1.66 m long shaft with four blades spaced at 0.3 m attached to the bottom (Figure 3-3). The bottom blades were attached 150 mm from the bottom. These blades were rotated at 180° to each other to ensure uniform mixing. The

upper end of shaft was attached to a motor via a gear used to control its speed and direction of rotation.

3.3.5 Air Line

An air line was attached to the sulphur vessel to ensure a constant pressure inside the vessel so that a constant flow rate could be maintained. The air line was attached to a regulator (Figure 3-4). During the test when molten sulphur and salt were in the sulphur vessel and the mixer was operating, the hole at top of the sulphur vessel was plugged with a cap and the air line was attached (Figure 3-5). The air pressure was maintained between 35 and 55 Pascal (N/m^2) during the test. The constant pressure allowed the mixture to be discharged at a uniform flow rate. Under the gravitational effect, i.e., without this applied pressure, a non-uniform flow was expected but also there was the possibility that sulphur would not flow out of vessel when the mixer was in operation.

3.3.6 Crane

The crane was used to lift the apparatus during the experiment to measure its mass. The load cell was attached to the data logger to monitor the mass flow rate during experiment. Figure 3-6 shows the crane and measuring system.

3.3.7 Platform

A discharge platform 1.80 m wide and 4 m in length was used to study the flow behavior of the sulphur during the experiments. It was made with steel sheets supported on concrete slabs (Figure 3-7). On the sides were steel troughs to catch the flowing sulphur (Figure 3-8). The flow pattern of the sulphur mixtures was observed during discharge onto this platform.

3.4 Materials

3.4.1 Sulphur

The sulphur used in the experimental program was obtained from Syncrude Canada Ltd in 50 kg (20 liter) metal pails.

3.4.2 Salt

The salt used in the experimental program was obtained from Syncrude in the form of brine. The brine was evaporated in a laboratory oven and the solid residue was then broken into smaller pieces. Sufficient brine was not available in large quantity because it was also used in the other research program conducted by Ding (2006). To study the flow properties, a large quantity of commercially available salt was used for this study. Salt obtained in blocks was provided by the Canada Salt Company Ltd. (CAS No. 7647-14-5). It has a specific gravity of 2.165 and a melting point of 801 °C.

The salt was received in blocks that were then crushed and sorted to the particle size distribution that was used in the research program carried out by Ding (2006). The comparison of the grain size distribution between these two research programs are shown in Figure 2-10 and 2-11.

3.5 Test Procedure

The tests were carried out in the following sequence.

1. Prior to the beginning of test, the platform was leveled to the required 2% slope in its longitudinal direction. Transverse leveling of platform was done across the length to eliminate any flow to the sides.
2. The sulphur was raised in temperature to above a 110 °C to form a liquid. It was melted in following two ways.

- Pails of sulphur were placed in the ventilated oven maintained at a temperature of 115°C. It required about 8 hours to melt the sulphur.
- Sulphur was removed from the metal pail, broken into small pieces and then heated at site with a tiger torch. In this case the temperature was not controlled and it was overheated in contact with the flame. Due to overheating the viscosity increases tremendously. It was then cooled to the required temperature by letting cool while being stirred occasionally (continuous stirring was not possible due to release of pungent fumes at temperatures above 160 °C).

3. The propane torch was put under the oil bath early in the morning to bring the oil to the required test temperature. Thermistors attached to the data logger measured the temperature of oil bath and inside the sulphur vessel.
4. Once the temperature in the sulphur reached the test temperature, the sulphur was poured into the vessel using a steel funnel. Pre-heated salt was then added to the vessel while the mixer kept the sulphur and salt well mixed.
5. The air line was then attached to the sulphur vessel and pressure was applied to ensure that the flow would occur at a uniform rate.
6. The whole apparatus was then lifted by the crane and the attached load cell measured the weight of the apparatus and sulphur when the discharge was started. The load cell monitored the change in mass of the sulphur during the test. This allows the mass flow rate of the discharge to be monitored continuously throughout the test.
7. Once the apparatus was lifted, the pipes attached to the sulphur vessel were heated to melt the solid sulphur from the previous test. During this process, the valve was opened to let the sulphur flow into a pail so that the overheated sulphur was collected.
8. Sulphur from the vessel is discharged onto the platform until the vessel is empty (Figure 3-9).

9. The profile of crystallized sulphur on the surface of platform was measured both in the longitudinal as well as the transverse direction. Samples were collected from random locations so that the salt concentration in sulphur could be measured. Figure 3-10 shows the salt and sulphur distribution after a discharge event.

3.5.1 Test configurations

The following tests were conducted during the research

- Test 1: Sulphur alone. Quantity of sulphur used was 58 kg.
- Test 2: 4% salt and 96% sulphur. Quantity of sulphur 69.68 kg and salt 2.71 kg.
- Test 3: 9% salt and 91% sulphur. Quantity of sulphur 41.19 kg and salt 3.62 kg
- Test 4: 15% salt and 85% sulphur. Quantity of sulphur 42.34 kg and salt 6.39 kg
- Test 5: Only sulphur. Quantity of sulphur was 42.33 kg
- Test 6: 20% salt and 80% sulphur. Quantity of sulphur 30 kg and salt 6 kg
- Test 7: 25% salt: test was not performed as the discharge pipes were blocked with a large quantity of salt.

3.6 Sampling and Insitu Measurement

The following measurements were taken during and after each test.

1. Air temperature
2. Temperature of flowing sulphur
3. Mass rate of flow.
4. Maximum flow length of the sulphur.

5. Surface profile of the sulphur after test with tape measurement.

Samples were collected from random locations within the deposited material after each test. These samples were analyzed to determine the percentage of salt and sulphur. This analysis was done in laboratory by mixing the solid sulphur and salt mixture with the distilled water. Initial EC value of distilled water was observed. This mixture was then filtered so that salt mixed in water was drained and only solid sulphur was left. EC value of this water was observed and if found greater then the initial value for the distilled water the procedure was repeated again till the filtered water gave the initial EC value. This ensures that there is no more salt present in the sulphur. Sulphur was then oven dried and weighed. These measurements were used to establish the concentration of salt within the sulphur samples collected at different locations within the deposit. Figure 3-11 shows the distribution of salt in the sulphur deposit.

3.7 Limitations and Assumptions

The experiment was performed with the following limitations and assumptions.

1. A narrow working range for the temperature was available: 115 °C to 160 °C. Heating was achieved using a propane torch and controlling the temperature was difficult. Pipes were heated with a propane torch to melt the sulphur left over from previous tests and this sometimes resulted in over heating of the pipes.
2. The platform was of limited dimensions. Due to hydraulic sorting the flow of slurry was diverted to the sides of the discharge area as discussed in section 2.3.5.
3. Unidirectional flow from a single discharge point was used in experiment. In the field multi-point or radial flow discharge may be used.
4. The grain size of salt was restricted to that used in environmental impact research by Ding (2006). In the experiments, commercially available salt was used due to limited quantity of brine available for the two research programs.

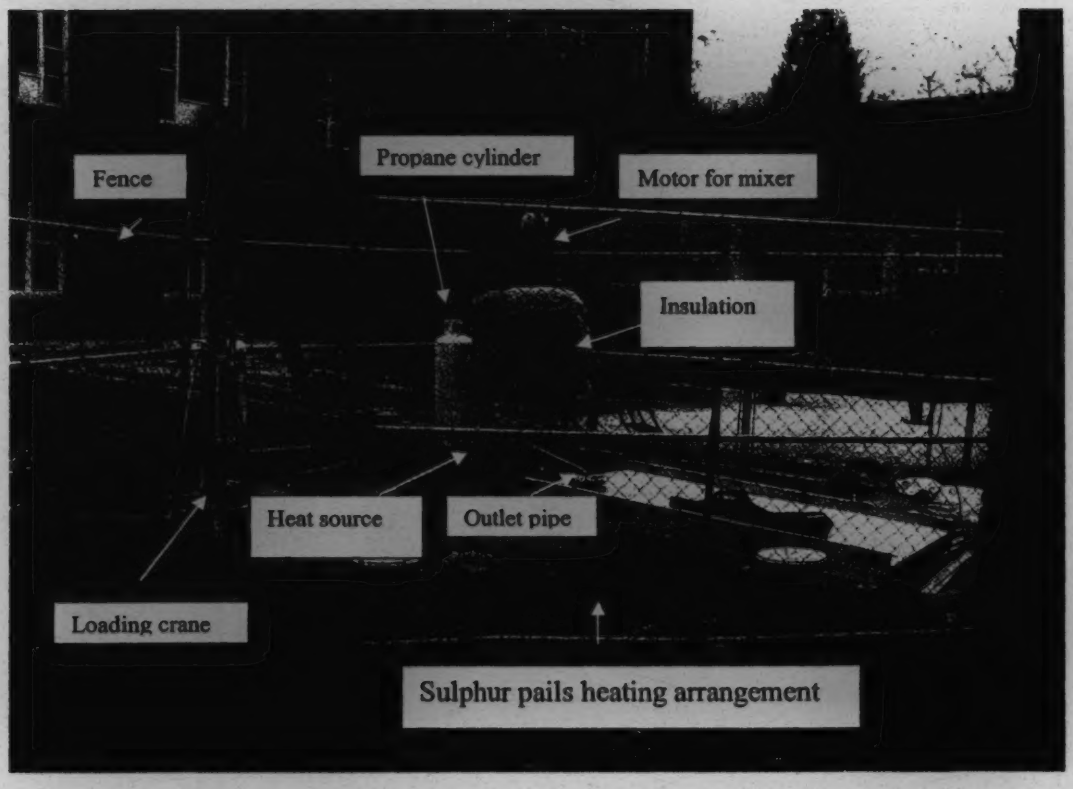

Figure 3-1 General apparatus layout

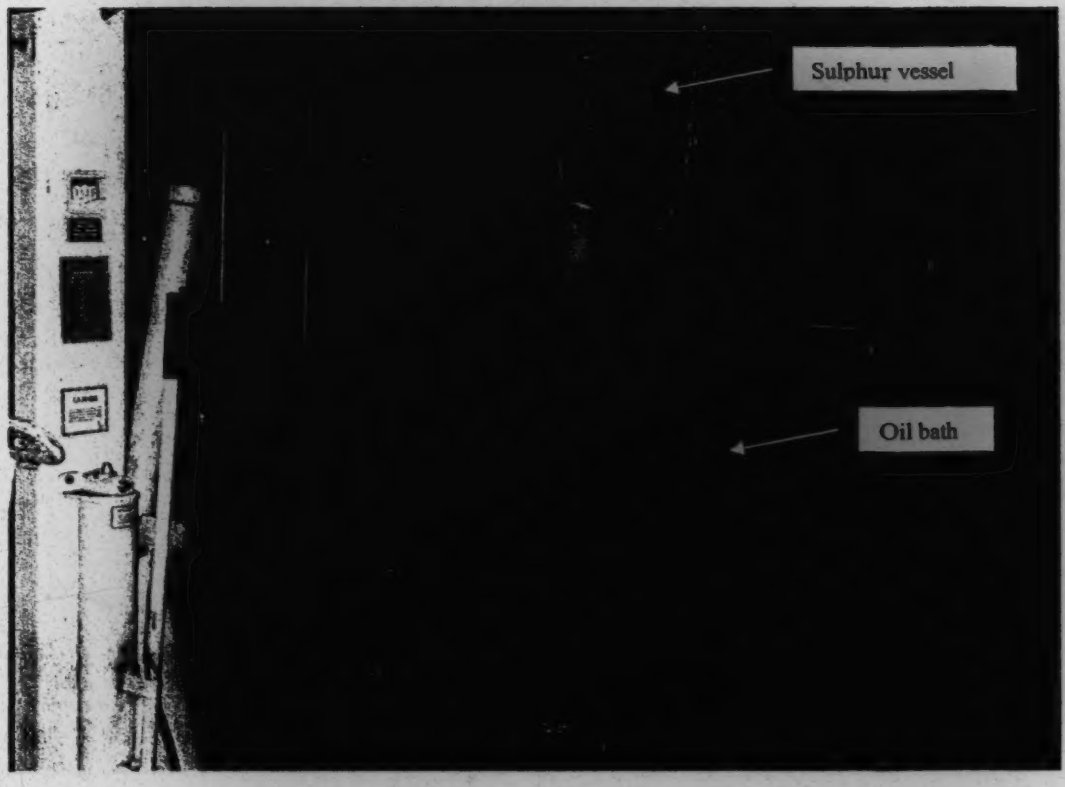

Figure 3-2 Oil bath and sulphur vessel

Figure 3-3 Mixer

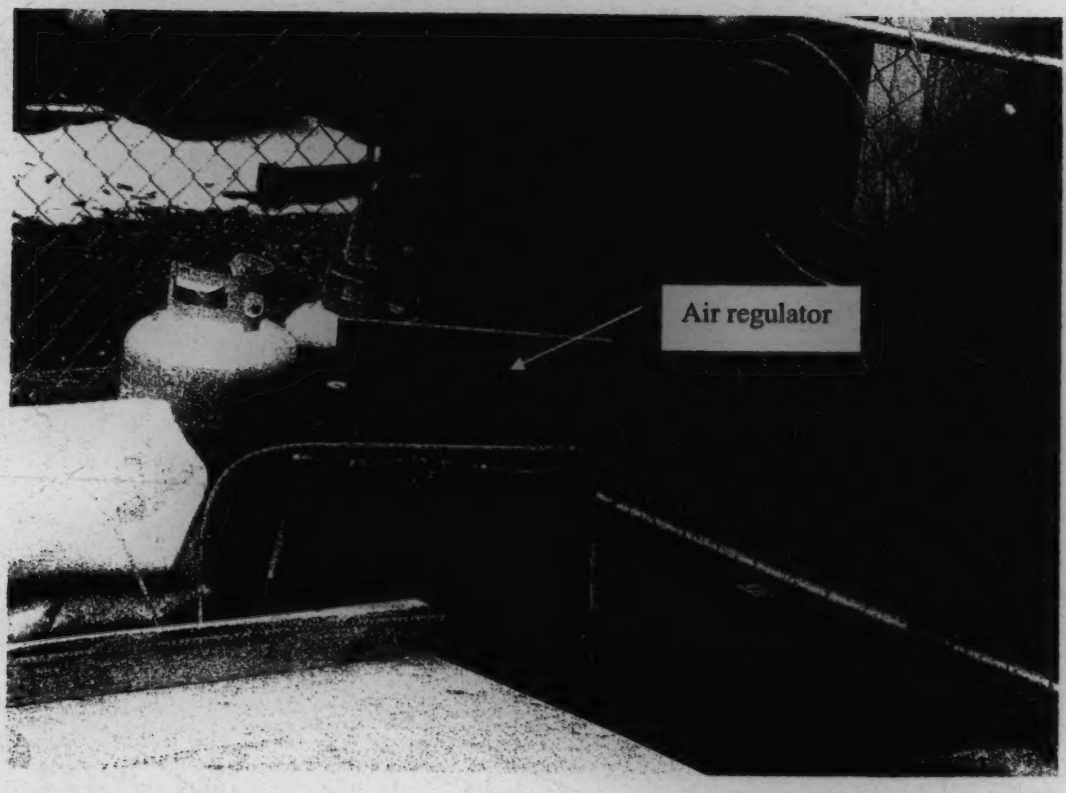

Figure 3-4 Air regulator

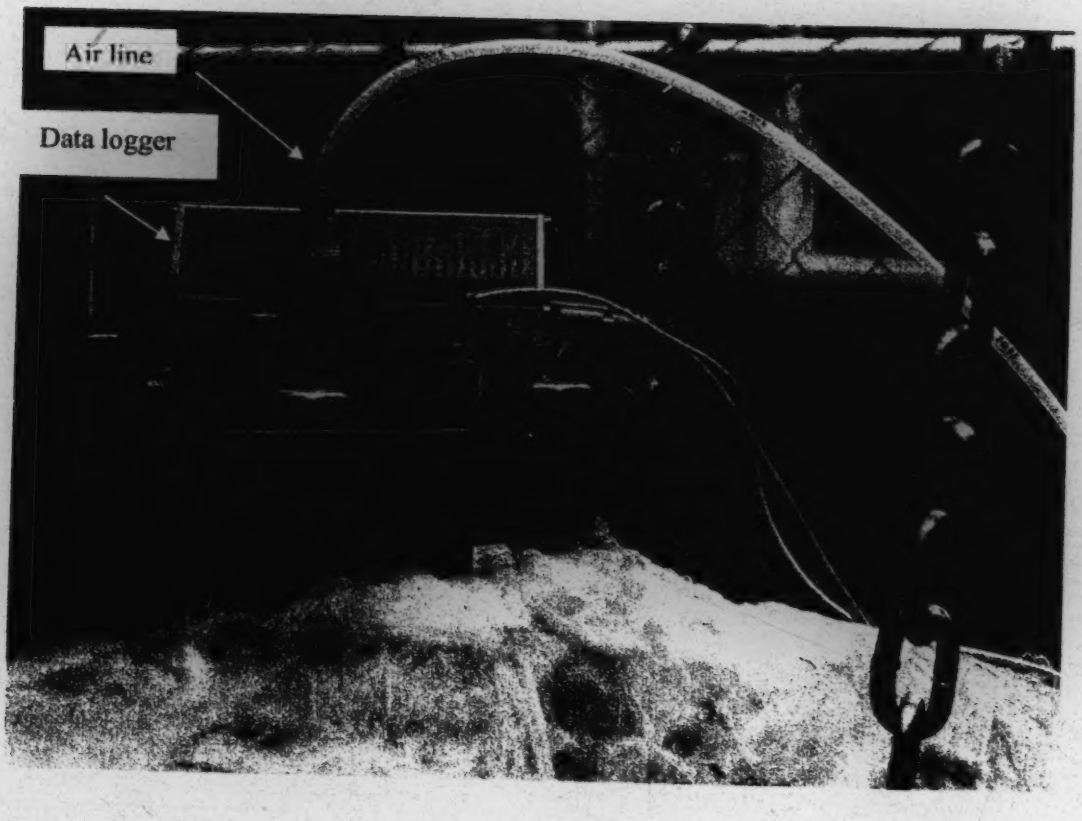

Figure 3-5 Air line and data logger

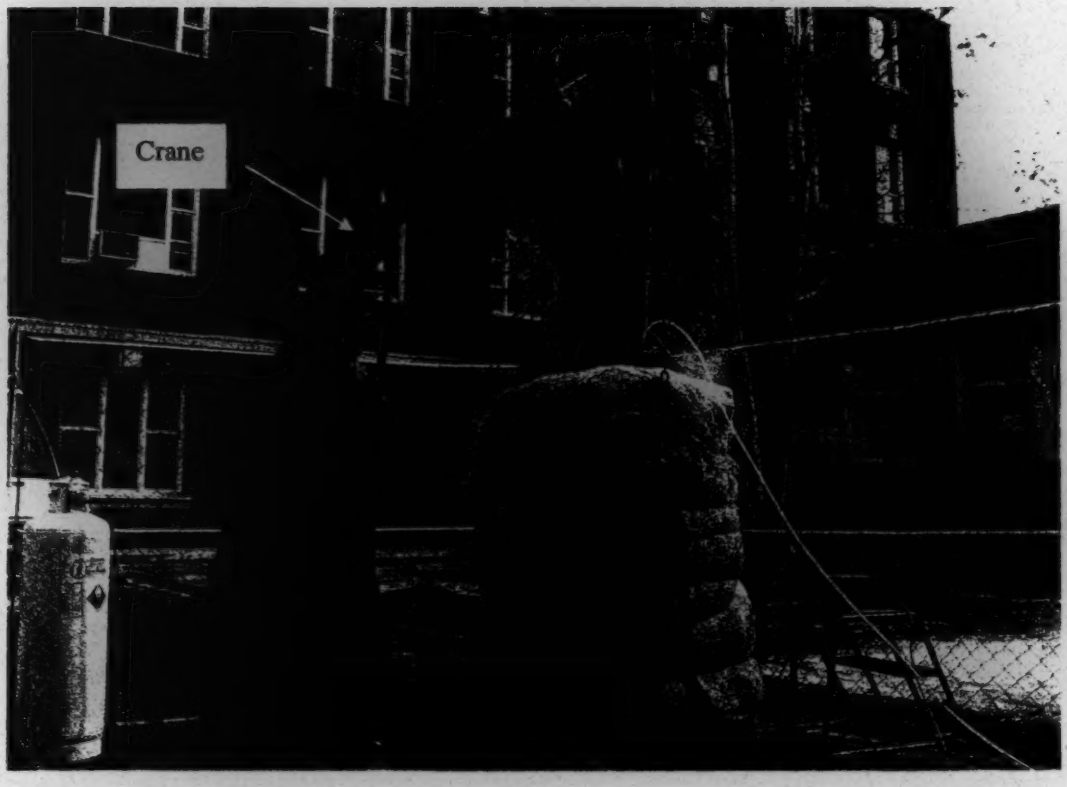

Figure 3-6 Apparatus hanging from crane

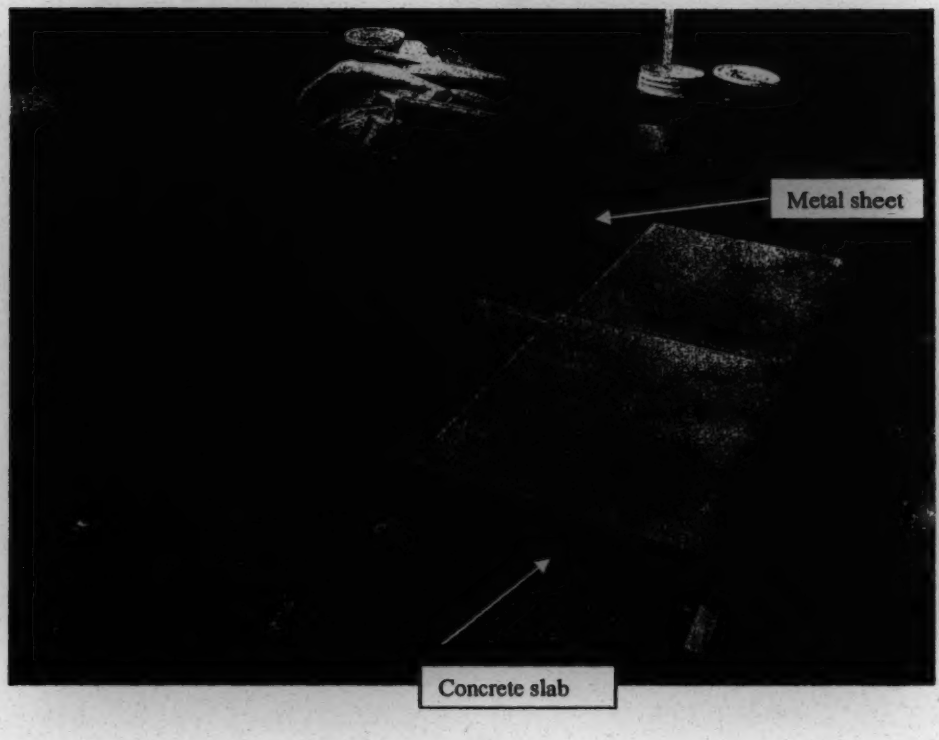

Figure 3-7 Platform during assembly

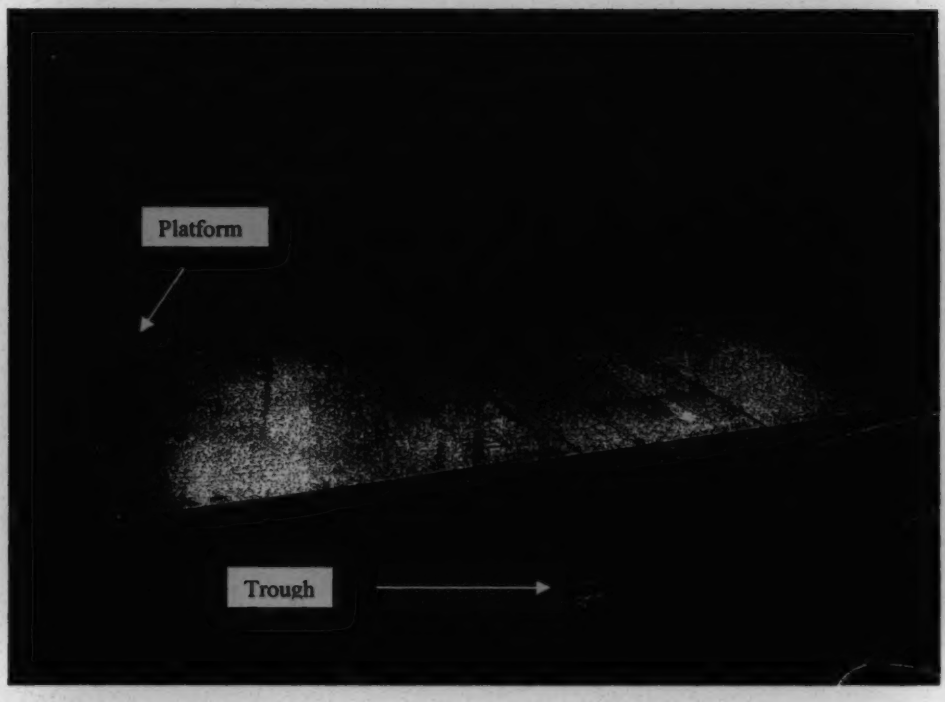

Figure 3-8 Platform

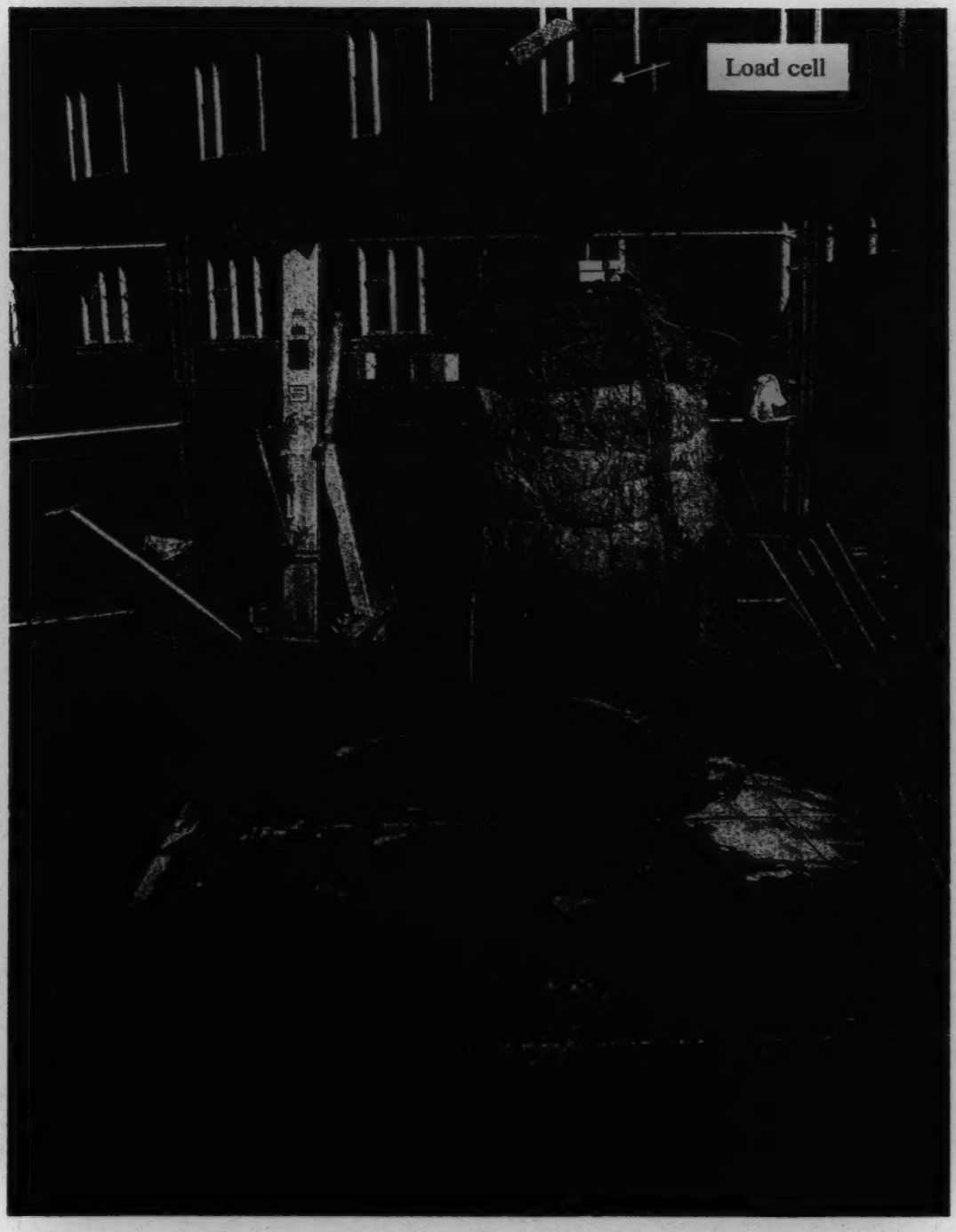

Figure 3-9 Fresh sulphur and salt mixture flowing over previously laid sulphur.

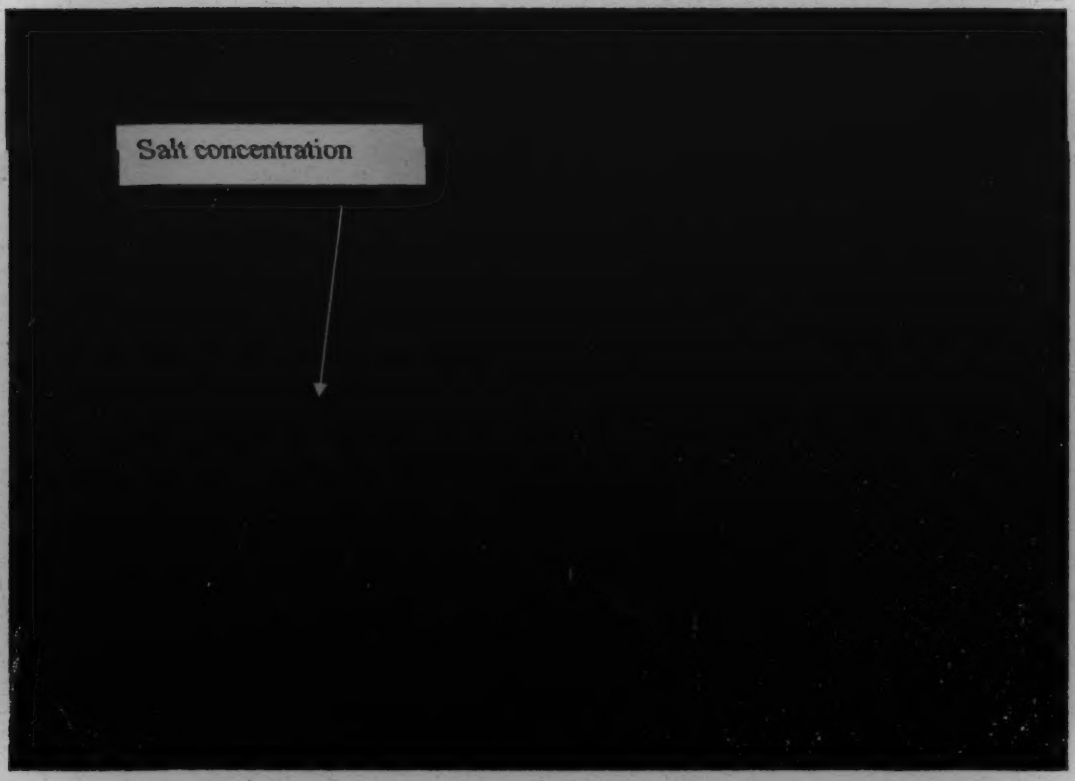

Figure 3-10 Distribution of salt in sulphur following discharge

Figure 3-11 Figure showing distribution of salt. Hydraulic sorting of salt is evident

4 RESULTS

This chapter begins by presenting the numerical results from the analytical model (Otto, 2002). The influence of sulphur discharge temperature, outside ambient air temperature and mass flow rate at the discharge outlet on the predicted significant length. Seven tests were performed using different discharge velocities, ambient temperature and salt concentrations, and the surface discharge profiles were observed. The first trial test was performed using only sulphur, and the fifth test was performed to verify the results of the first test. The remaining five tests were performed with varying mass concentrations of salt within the sulphur.

4.1 Sulphur Discharge Tests (1 and 5)

Tests one and five were performed to calculate the significant length of only sulphur. Tests with sulphur alone were performed for two reasons

- To evaluate the performance of the test setup and procedure
- To compare the numerical model results with those from the tests.

Test 1 discharged 58 kg of molten sulphur, test 5 used 42 kg of sulphur. The sulphur temperature was 120 °C to observe the flow behavior of elemental sulphur. The outside air / platform temperature was measured at 4.8 and 15.8 °C for tests 1 and 5 respectively and the significant lengths measured were 3.2 m and 3.7 m. The mass rate of flow was 13.7 and 15.6 kg/s-m for tests 1 and 5 respectively.

Prediction of significant length results obtained from the numerical model developed by Otto (2002), and the significant lengths calculated at varying discharge sulphur temperatures between 120 °C and 140 °C are presented in Table 4-1 to 4-4.

In the numerical model, outside air / platform temperatures were analyzed for the temperature range of -25 °C to 25 °C. Mass flow rates in the range of 3 to 24 kg/s-m were modeled. The results are summarized in Table 4-1 to 4-4 and shown graphically in

Figures 4-1 to 4-7. Figure 4-2 to 4-4 illustrates the trend shown in Figure 4-1 for constant sulphur discharge temperatures of 120, 130 and 140 °C. The significant length increases as the mass flow rate increases. Figure 4-5 to 4-7 illustrate that under a constant mass flow rate and a given sulphur discharge temperature the significant length increases as the ambient air temperature increases.

The flow pattern of sulphur only (Test 1) is shown in Figure 4-9. The flow of molten sulphur was observed to be almost evenly distributed both in the longitudinal and transverse direction. The thickness of the crystallized sulphur was observed to be about 2 mm. The flow behavior of sulphur was observed to be dependent on its internal heat loss mechanism only.

The two tests on sulphur only were performed at outside temperatures of 4.8 °C and 15 °C. The comparison shows that the difference between the test results and predicted significant length is less when the outside temperature is warmer (Figure 4-8). The significant lengths obtained using the numerical model are 13 % and 5 % greater than the test measured lengths for ambient air temperatures of 4.8 °C and 15 °C respectively. The difference between the significant length and numerical model length is not considered significant. Hence it was assumed that the tests to be carried out with addition of salt within sulphur will also have this range of deviation when the test results are compared to the numerical model predicted significant lengths.

4.2 Sulphur mixed with Salt

Five tests were performed with varying salt concentrations (4%, 9%, 15%, 20% and 25%) in the sulphur. All the tests were carried out at 120 °C temperature to compare the flow behavior with different salt concentrations. The flow of 25% salt mixed with sulphur could not be performed due to a blockage of the discharge pipes of the experimental apparatus.

The flow pattern (profile) of salt sulphur mixture was recorded after the each test. The measurements of these flow patterns were carried out using a tape measure in the longitudinal direction (significant length) and transverse direction on the test platform. The discharge outlet was considered as the reference discharge point. Samples were collected at random locations to determine the quantity of salt deposited with the sulphur. Figures 4-10 to 4-13 show the flow profiles for different salt concentrations contained in the sulphur.

4.2.1 Sulphur with 4% Salt (Test #2)

Flow for this test took place over the crystallized sulphur layer formed during test 1. The quantity of sulphur used was 69.68 kg and the quantity of salt used was 2.71 kg. The outside air / platform temperature was 14 °C. The mass flow rate observed during this test was 16.5 kg/s-m. The significant length measured in the test was 2.6 m. Figure 4-10 shows the flow pattern observed during this test. The observed flow behavior was similar to that of the sulphur only (Test #1). The flow was evenly distributed both in its longitudinal and transverse directions.

4.2.2 Sulphur with 9% Salt (Test #3)

Flow for this took place over the crystallized sulphur layer formed during Test #2. The quantity of sulphur used was 41.2 kg and the quantity of salt used was 3.6 kg. The test was conducted with the outside air / platform temperature of 12 °C, and the flow rate of 15 kg/s-m. The measured significant length in the test was 2.8 m (Figure 4-11). The flow behavior was distributed evenly within the initial 1.5 m of the deposit. Pockets of coarse salt particles were observed in this portion of flow. Due to these salt pockets the later flow was channelized (braided nature) between 1.5 m to the end point at 2.7 m. This channelized flow was concentrated towards the left side of the deposition area on the platform.

4.2.3 Sulphur with 15% Salt (Test #4)

Flow for this mixture took place over the crystallized sulphur layer formed during Test #3. The quantity of sulphur used was 42.3 kg and the quantity of salt used was 6.39 kg. The test was conducted with the outside air / platform temperature of 18 °C, and a flow rate of 13 kg/s-m. The measured significant length in the test was 2.1 m (Figure 4-12). The flow behavior was distributed evenly in the initial 1 m length. High concentration of coarse salt particles was observed in this initial 1 m flow length. The more distance portion of flow was dictated by the salt concentrated pockets near the discharge outlet which gave rise to a highly braided nature of flow. The crystallized sulphur towards the end of the flow pattern was devoid of coarser salt particles giving rise to finger like projections of the sulphur in this region.

4.2.4 Sulphur with 20% Salt (Test #6)

This test was performed with 20% salt content mixed in sulphur and the flow took place over the sulphur layer formed during test 5. The quantity of sulphur used was 30 kg and the quantity of salt used was 6 kg. The test was conducted with the outside air / platform temperature of 8 °C, and a flow rate of 16 kg/s-m. The measured significant length in the test was 1.4 m (Figure 4-13). The flow behavior was distributed primarily in the transverse directions from the initial discharge point. The significant length was reduced due to the diversion of flow in the transverse direction. A high concentration of coarse salt particles was observed in the initial 0.75 m flow length (close to the discharge outlet). Channelized flow was observed in the latter flow length due to the high concentration of salt in the initial portion. It was observed that due to transverse flow the sulphur was directed off the platform into the trough rather then flowing longitudinally.

In Table 4-5 it is observed that in the test with 4% salt concentration the significant length achieved was 260 cm and flow was dispersed over the entire platform. At 9% salt concentration the significant length was almost the same but the flow was dispersed to 100% of the platform width in the first one third and in the later length it occupied 30% of the platform width. In case of 15% salt concentration, the flow was

dispersed to full width in the first one third portion, in the second one third portion it occupied 70% width of the platform, and in the last one third it occupied only the 30% of the width of platform. In the case of 20% salt concentration the flow was dispersed over the entire platform to the length the sulphur flowed. More spreading of the flow across the platform is observed when the concentration of salt mixed with the molten sulphur increased.

4.3 Salt Content Distribution

The flow behavior observed during these tests was influenced by the concentration of salt. In order to determine the concentration of salt in the sulphur during placement, several samples were collected at random locations across the platform and were tested in the laboratory. Salt concentrations were measured at different locations across the platform as discussed in section 3.6. Figures 4-14 and 4-16 show the salt concentrations measured at various locations in the solidified sulphur salt mass.

For the test with 4% salt, salt concentration values ranged from 2 % to 19 % at various locations. The salt concentration values normalized to the initial salt concentration in this case ranges from 0.5 to 4.75 (Figure 4-15). Generally higher concentrations were observed near the discharged outlet and the concentration values decreased as the distance from discharge outlet.

For the test with 20% salt mix, salt concentration values ranged from 2 to 40 %. The salt concentration values normalized to the initial salt concentration in this case ranges from 0.1 to 2.0 (Figure 4-17). Higher concentrations of salt were found near the discharge outlet, they decreased as the flow progressed toward the outer edges. Both figures show that the pattern of salt concentration near the discharge outlet is such that maximum salt concentration is found within the initial first third of the significant length for the particular mixture.

Another significant factor that will affect the salt settling behavior is its specific gravity. Ding (2006) reported the specific gravity of salt from the oil sands brine as 2.63. The salt used in this study was commercially available salt with specific gravity of 2.17. One possible impact of a lower specific gravity will be the reduced tendency to settle and possibly greater flow lengths due to lower density of the salt particles. As the actual oil sands salt is heavier, the particles will settle faster from sulphur therefore the salt will settle closer to discharge than the salt used in these tests.

Initial Temp of Sulphur = 120°C

Mass Rate of Flow	Air Temp	Air Temp	Length 1	Length 2	Significant Length
kg/sec m	°C	°K	m	m	m
3	4.8	277.8	0.0790	0.753	0.83
7	4.8	277.8	0.2310	1.721	1.95
10	4.8	277.8	0.3650	2.433	2.80
13	4.8	277.8	0.5100	3.13	3.64
17	4.8	277.8	0.7150	4.056	4.77
20	4.8	277.8	0.8770	4.741	5.62
24	4.8	277.8	1.1000	5.649	6.75
3	10	283	0.0800	0.774	0.85
7	10	283	0.2450	1.766	2.01
10	10	283	0.3850	2.496	2.88
13	10	283	0.5380	3.216	3.75
17	10	283	0.7520	4.166	4.92
20	10	283	0.9200	4.871	5.79
24	10	283	1.1600	5.803	6.96
3	-10	263	0.0680	0.704	0.77
7	-10	263	0.2030	1.605	1.81
10	-10	263	0.3180	2.266	2.58
13	-10	263	0.4450	2.918	3.36
17	-10	263	0.6220	3.777	4.40
20	-10	263	0.7620	4.415	5.18
24	-10	263	0.9570	5.258	6.22
3	15	288	0.0860	0.7949	0.88
7	15	288	0.2580	1.814	2.07
10	15	288	0.4050	2.564	2.97
13	15	288	0.5650	3.304	3.87
17	15	288	0.7950	4.28	5.08
20	15	288	0.9750	5.005	5.98
24	15	288	1.2250	5.964	7.19
3	-15	258	0.0660	0.689	0.76
7	-15	258	0.1950	1.57	1.77
10	-15	258	0.3060	2.217	2.52
13	-15	258	0.4250	2.855	3.28
17	-15	258	0.5960	3.695	4.29
20	-15	258	0.7300	4.318	5.05
24	-15	258	0.9170	5.141	6.06

Table 4-1 Calculated significant length at sulphur initial temperature of 120°C

Initial Temp of Sulphur = 120°C

Mass Rate of Flow	Air Temp	Air Temp	Length 1	Length 2	Significant Length
kg/sec m	°C	°K	m	m	m
3	25	298	0.0950	0.841	0.94
7	25	298	0.2900	1.921	2.21
10	25	298	0.4550	2.716	3.17
13	25	298	0.6350	3.501	4.14
17	25	298	0.8890	4.538	5.43
20	25	298	1.0950	5.308	6.40
24	25	298	1.3750	6.326	7.70
3	-20	253	0.0635	0.6758	0.74
7	-20	253	0.1870	1.538	1.73
10	-20	253	0.2935	2.171	2.46
13	-20	253	0.4080	2.7951	3.20
17	-20	253	0.5720	3.616	4.19
20	-20	253	0.7000	4.226	4.93
24	-20	253	0.8790	5.031	5.91
3	20	293	0.0920	0.8171	0.91
7	20	293	0.2730	1.865	2.14
10	20	293	0.4290	2.637	3.07
13	20	293	0.5990	3.399	4.00
17	20	293	0.8400	4.404	5.24
20	20	293	1.0310	5.151	6.18
24	20	293	1.2960	6.138	7.43
3	-25	248	0.0600	0.662	0.72
7	-25	248	0.1800	1.506	1.69
10	-25	248	0.2810	2.127	2.41
13	-25	248	0.3920	2.738	3.13
17	-25	248	0.5480	3.542	4.09
20	-25	248	0.6720	4.139	4.81
24	-25	248	0.8430	4.927	5.77

Initial Temp of Sulphur = 130°C

Mass Rate of Flow	Air Temp	Air Temp	Length 1	Length 2	Significant Length
kg/sec m	°C	°K	m	m	m
3.00	5	278	0.1980	0.755	0.95
7.00	5	278	0.5850	1.722	2.31
10.00	5	278	0.9195	2.435	3.35
13.00	5	278	1.2800	3.137	4.42
17.00	5	278	1.7950	4.062	5.86
20.00	5	278	2.2000	4.749	6.95
24.00	5	278	2.7650	5.657	8.42
3.00	10	283	0.2070	0.7744	0.98
7.00	10	283	0.6130	1.765	2.38
10.00	10	283	0.9640	2.496	3.46
13.00	10	283	1.3425	3.216	4.56
17.00	10	283	1.8830	4.166	6.05
20.00	10	283	2.3100	4.871	7.18
24.00	10	283	2.9020	5.803	8.71
3.00	-10	263	0.1740	0.705	0.88
7.00	-10	263	0.5120	1.606	2.12
10.00	-10	263	0.8040	2.268	3.07
13.00	-10	263	1.1190	2.921	4.04
17.00	-10	263	1.5680	2.782	5.35
20.00	-10	263	1.9210	4.418	6.34
24.00	-10	263	2.4120	5.262	7.67
3.00	15	288	0.2180	0.795	1.01
7.00	15	288	0.6440	1.815	2.46
10.00	15	288	1.0140	2.566	3.58
13.00	15	288	1.4130	3.306	4.72
17.00	15	288	1.9820	4.284	6.27
20.00	15	288	2.4310	5.009	7.44
24.00	15	288	3.0560	5.969	9.03
3.00	-15	258	0.1670	0.69	0.86
7.00	-15	258	0.4920	1.571	2.06
10.00	-15	258	0.7720	2.219	2.99
13.00	-15	258	1.0740	2.857	3.93
17.00	-15	258	1.5030	3.697	5.20
20.00	-15	258	1.8420	4.321	6.16
24.00	-15	258	2.3120	5.145	7.46

Table 4-2 Calculated significant length at sulphur initial temperature of 130°C

Initial Temp of Sulphur = 130°C

Mass Rate of Flow	Air Temp	Air Temp	Length 1	Length 2	Significant Length
kg/sec m	°C	°K	m	m	m
3.00	20	293	0.2290	0.8172	1.05
7.00	20	293	0.6780	1.8662	2.54
10.00	20	293	1.0680	2.637	3.71
13.00	20	293	1.4880	3.399	4.89
17.00	20	293	2.0880	4.402	6.49
20.00	20	293	2.5630	5.15	7.71
24.00	20	293	3.2220	6.134	9.36
3.00	-20	253	0.1600	0.676	0.84
7.00	-20	253	0.4730	1.539	2.01
10.00	-20	253	0.7410	2.172	2.91
13.00	-20	253	1.0310	2.797	3.83
17.00	-20	253	1.4430	3.619	5.06
20.00	-20	253	1.7680	4.229	6.00
24.00	-20	253	2.2190	5.035	7.25
3.00	25	298	0.2420	0.841	1.08
7.00	25	298	0.7150	1.921	2.64
10.00	25	298	1.1270	2.716	3.84
13.00	25	298	1.5730	3.502	5.08
17.00	25	298	2.2080	4.538	6.75
20.00	25	298	2.7100	5.308	8.02
24.00	25	298	3.4070	6.326	9.73
3.00	-25	248	0.1550	0.662	0.82
7.00	-25	248	0.4540	1.507	1.96
10.00	-25	248	0.7130	2.127	2.84
13.00	-25	248	0.9910	2.736	3.73
17.00	-25	248	1.3870	3.542	4.93
20.00	-25	248	1.6990	4.139	5.84
24.00	-25	248	2.1310	4.926	7.06

Initial Temp of Sulphur = 140°C

Mass Rate of Flow	Air Temp	Air Temp	Length 1	Length 2	Significant Length
kg/sec m	°C	°K	m	m	m
3.00	5	278	0.29	0.76	1.04
7.00	5	278	0.85	1.73	2.58
10.00	5	278	1.34	2.44	3.77
13.00	5	278	1.86	3.14	5.00
17.00	5	278	2.61	4.07	6.68
20.00	5	278	3.20	4.74	7.94
24.00	5	278	4.02	5.66	9.68
3.00	10	283	0.30	0.78	1.08
7.00	10	283	0.89	1.77	2.66
10.00	10	283	1.40	2.50	3.90
13.00	10	283	1.95	3.22	5.17
17.00	10	283	2.73	4.17	6.91
20.00	10	283	3.35	4.88	8.23
24.00	10	283	4.21	5.81	10.02
3.00	-10	263	0.25	0.71	0.96
7.00	-10	263	0.75	1.61	2.36
10.00	-10	263	1.18	2.27	3.45
13.00	-10	263	1.64	2.92	4.56
17.00	-10	263	2.29	3.78	6.07
20.00	-10	263	2.81	4.42	7.23
24.00	-10	263	3.52	5.27	8.79
3.00	15	288	0.32	0.80	1.11
7.00	15	288	0.93	1.82	2.75
10.00	15	288	1.47	2.57	4.04
13.00	15	288	2.05	3.31	5.35
17.00	15	288	2.87	4.29	7.16
20.00	15	288	3.52	5.01	8.53
24.00	15	288	4.42	5.97	10.39
3.00	-15	258	0.24	0.69	0.93
7.00	-15	258	0.72	1.57	2.29
10.00	-15	258	1.13	2.22	3.34
13.00	-15	258	1.57	2.86	4.43
17.00	-15	258	2.20	3.70	5.90
20.00	-15	258	2.69	4.32	7.02
24.00	-15	258	3.38	5.15	8.53

Table 4-3 Calculated significant length at sulphur initial temperature of 140°C

Initial Temp of Sulphur = 140°C

Mass Rate of Flow	Air Temp	Air Temp	Length 1	Length 2	Significant Length
kg/sec m	°C	°K	m	m	m
3.00	20	293	0.33	0.82	1.15
7.00	20	293	0.98	1.87	2.85
10.00	20	293	1.54	2.64	4.18
13.00	20	293	2.15	3.41	5.56
17.00	20	293	3.02	4.41	7.43
20.00	20	293	3.70	5.16	8.86
24.00	20	293	4.66	6.15	10.80
3.00	-20	253	0.24	0.68	0.91
7.00	-20	253	0.69	1.54	2.23
10.00	-20	253	1.09	2.17	3.26
13.00	-20	253	1.51	2.80	4.31
17.00	-20	253	2.11	3.62	5.73
20.00	-20	253	2.59	4.23	6.82
24.00	-20	253	3.25	5.03	8.28
3.00	25	298	0.35	0.84	1.19
7.00	25	298	1.03	1.93	2.96
10.00	25	298	1.63	2.72	4.35
13.00	25	298	2.27	3.51	5.78
17.00	25	298	3.18	4.55	7.73
20.00	25	298	3.90	5.32	9.22
24.00	25	298	4.91	6.34	11.25
3.00	-25	248	0.23	0.66	0.89
7.00	-25	248	0.67	1.51	2.18
10.00	-25	248	1.05	2.13	3.18
13.00	-25	248	1.45	2.74	4.20
17.00	-25	248	2.03	3.55	5.58
20.00	-25	248	2.49	4.14	6.63
24.00	-25	248	3.13	4.93	8.06

Mass Flow rate kg/sec m	3	7	10	13	17	20	24	
Temp (k)	Significant Length (m)							
-25	0.72	1.686	2.408	3.13	4.09	4.811	5.77	Temperature = 120 °C
-20	0.7393	1.725	2.4645	3.2031	4.188	4.926	5.91	
-15	0.76	1.77	2.52	3.28	4.29	5.05	6.06	
-10	0.772	1.808	2.584	3.363	4.399	5.177	6.215	
4.8	0.832	1.952	2.798	3.64	4.771	5.618	6.749	
10	0.854	2.011	2.881	3.754	4.918	5.791	6.963	
15	0.8809	2.072	2.969	3.869	5.075	5.98	7.189	
20	0.9091	2.138	3.066	3.998	5.244	6.182	7.434	
25	0.936	2.211	3.171	4.136	5.427	6.403	7.701	
Mass Flow rate kg/sec m	3	7	10	13	17	20	24	
Temp (k)	Significant Length (m)							
-25	0.817	1.961	2.84	3.727	4.929	5.838	7.057	Temperature = 130 °C
-20	0.836	2.012	2.913	3.828	5.062	5.997	7.254	
-15	0.857	2.063	2.991	3.931	5.2	6.163	7.457	
-10	0.879	2.118	3.072	4.04	5.348	6.339	7.674	
4.8	0.953	2.307	3.3545	4.417	5.857	6.949	8.422	
10	0.9814	2.378	3.46	4.5585	6.049	7.181	8.705	
15	1.013	2.459	3.58	4.719	6.266	7.44	9.025	
20	1.0462	2.5442	3.705	4.887	6.49	7.713	9.356	
25	1.083	2.636	3.843	5.075	6.746	8.018	9.733	
Mass Flow rate kg/sec m	3	7	10	13	17	20	24	
Temp (k)	Significant Length (m)							
-25	0.8904	2.177	3.176	4.196	5.581	6.634	8.058	Temperature = 140 °C
-20	0.912	2.232	3.26	4.309	5.734	6.82	8.278	
-15	0.934	2.292	3.343	4.429	5.898	7.017	8.528	
-10	0.96	2.356	3.445	4.555	6.07	7.225	8.787	
4.8	1.044	2.575	3.772	5	6.675	7.941	9.675	
10	1.076	2.659	3.9	5.17	6.906	8.228	10.02	
15	1.111	2.749	4.036	5.354	7.156	8.528	10.392	
20	1.149	2.848	4.183	5.556	7.428	8.86	10.802	
25	1.191	2.958	4.347	5.775	7.727	9.22	11.245	

Table 4-4 Calculated significant length at different air and sulphur temperature

%age of salt concentration in sulphur	Total observed Significant Length (cm)	%age of platform width covered in the initial one third of significant length	%age of platform width covered in the middle third of significant length	%age of platform width covered in the last one third of significant length
4%	260	100 %	100 %	100 %
9%	277	100 %	30%	30%
15%	208	100 %	70%	30%
20%	140	100 %	100 %	100 %

Table 4-5 Percentage of Transverse flow along the significant length

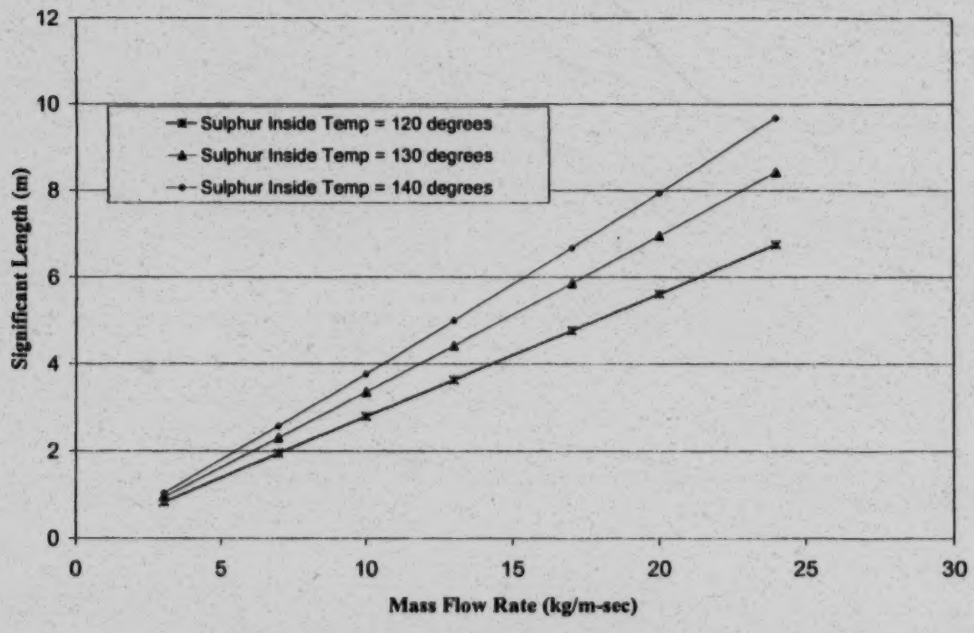

Figure 4-1 Calculated significant length at varying sulphur discharge temperature Tair = 4.8 °C

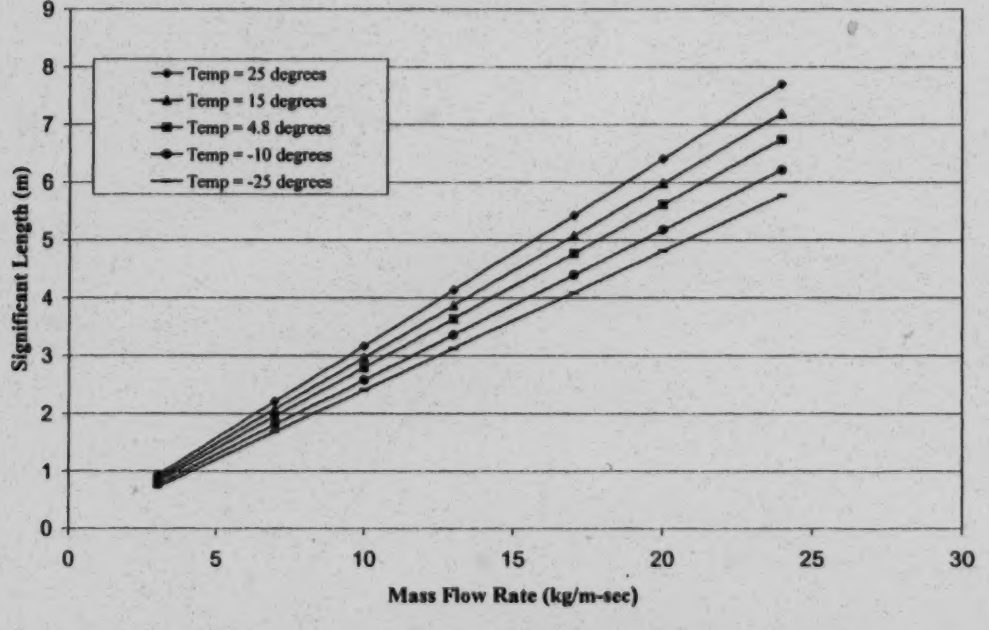

Figure 4-2 Calculated significant length at varying mass flow rates and Tair with initial temp 120°C

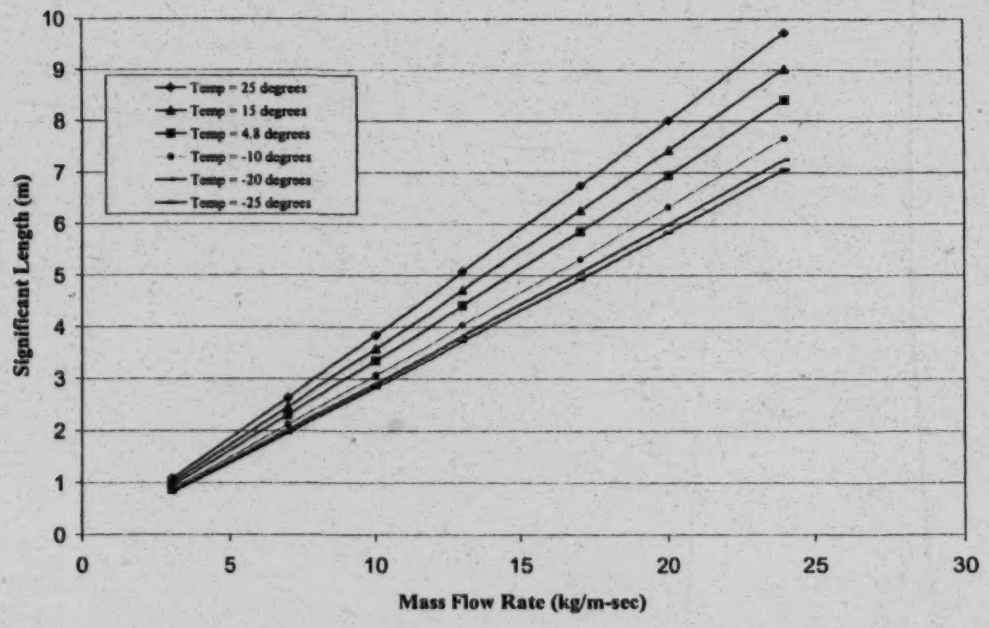

Figure 4-3 Comparison of significant length at varying mass flow rates and Tair with initial temp 130°C

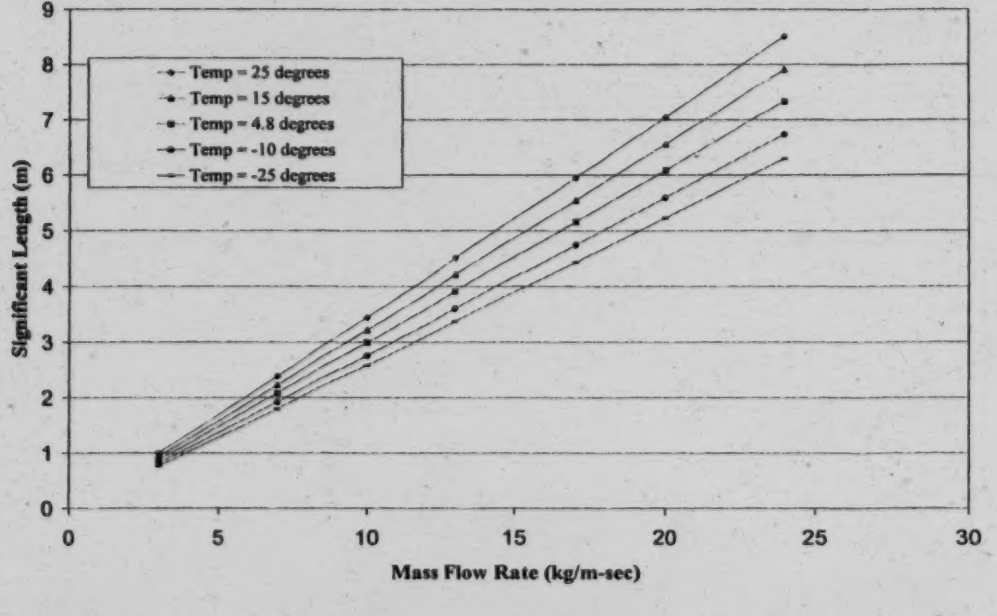

Figure 4-4 Comparison of significant length at varying mass flow rates and Tair with initial temp 140°C

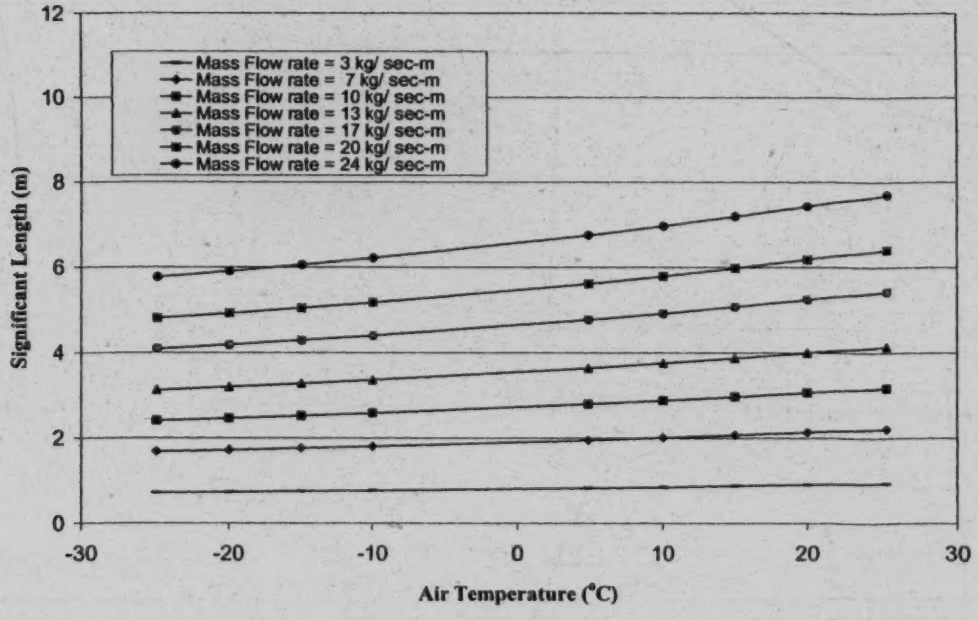

Figure 4-5 Comparison of significant length at varying temperature with initial temp 120°C

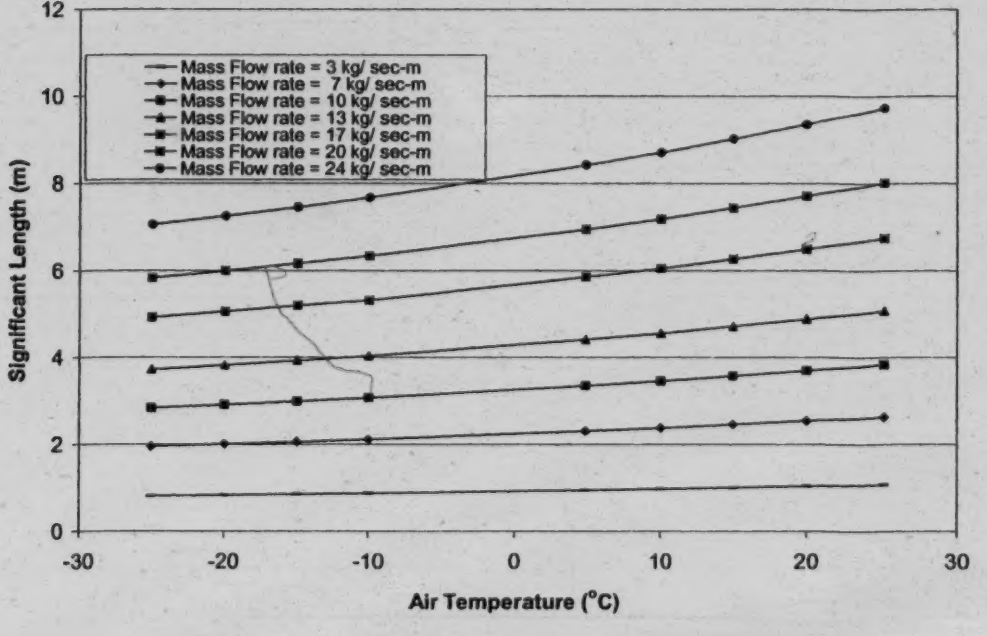

Figure 4-6 Comparison of significant length at varying temperature with initial temp 130°C

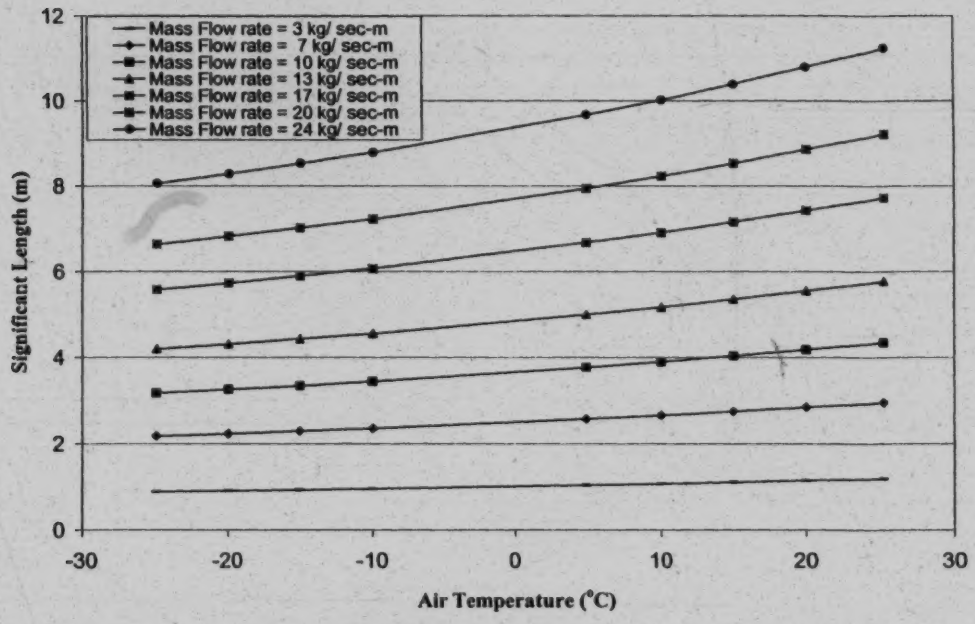

Figure 4-7 Comparison of significant length at varying temperature with initial temp 140°C

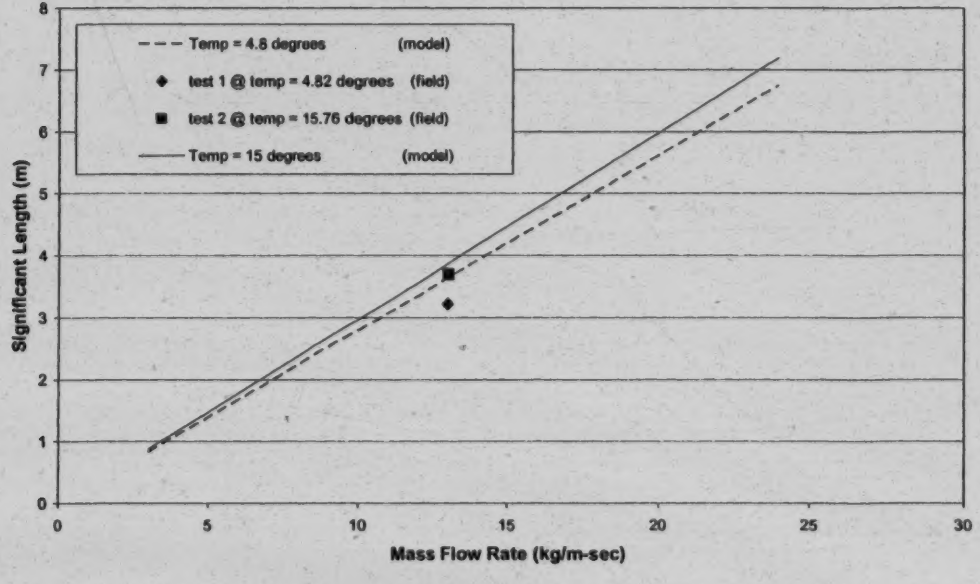

Figure 4-8 Comparison of field and numerical model data

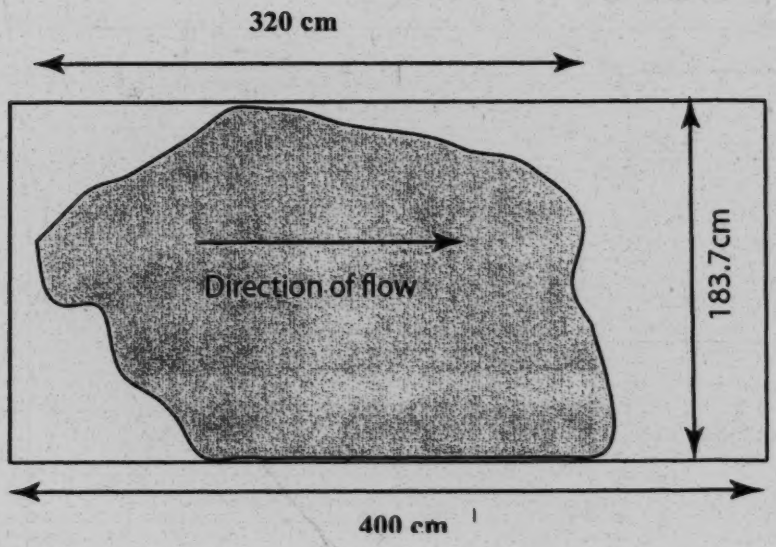

Figure 4-9 Flow profile with sulphur only (Test 1)

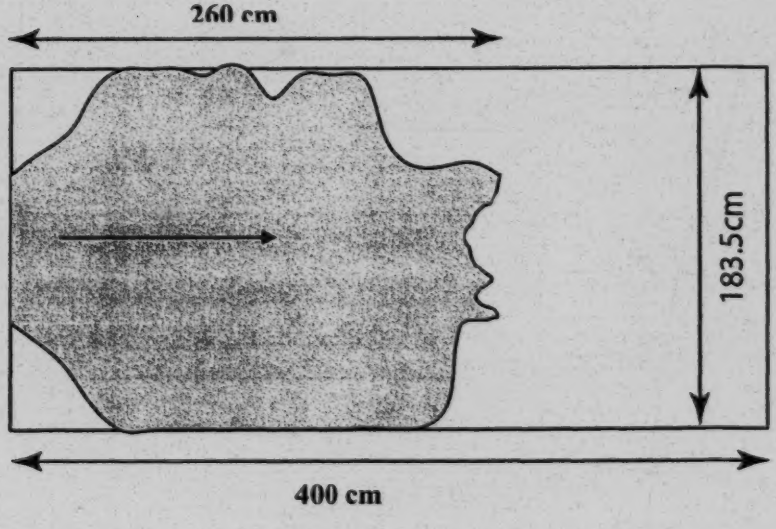

Figure 4-10 Flow profile with salt concentration of 4%

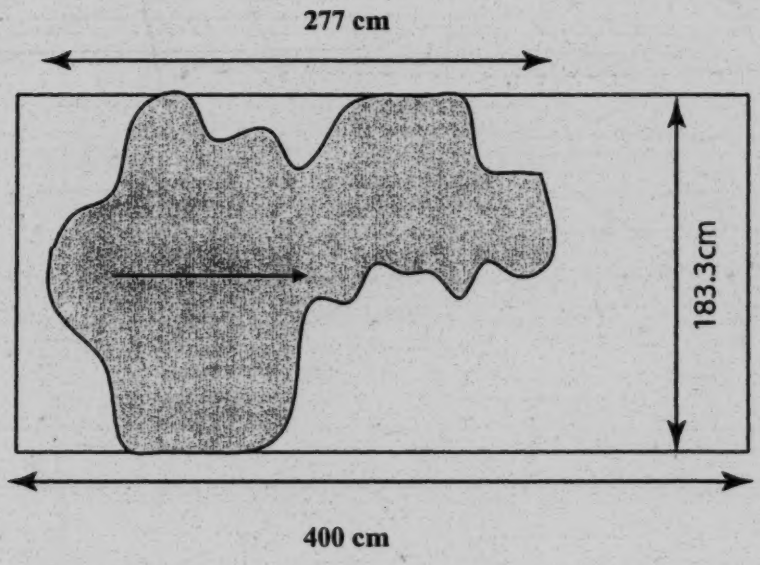

Figure 4-11 Flow profile with salt concentration of 9% salt

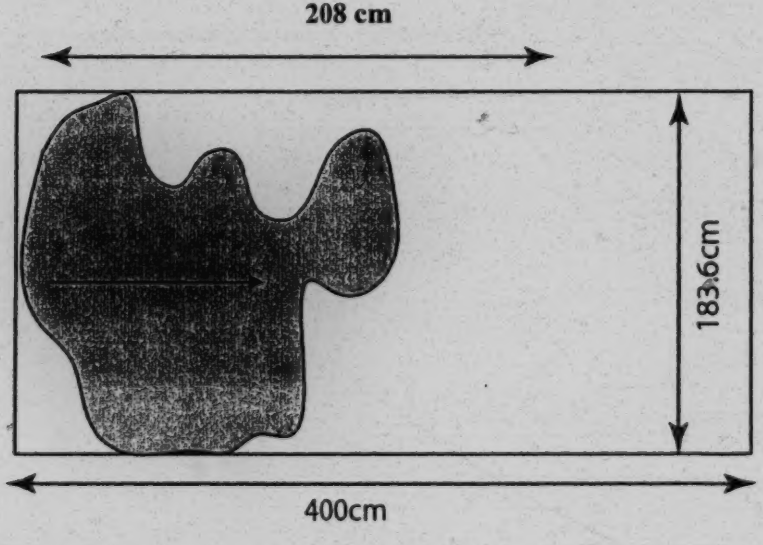

Figure 4-12 Flow profile with salt concentration of 15% salt

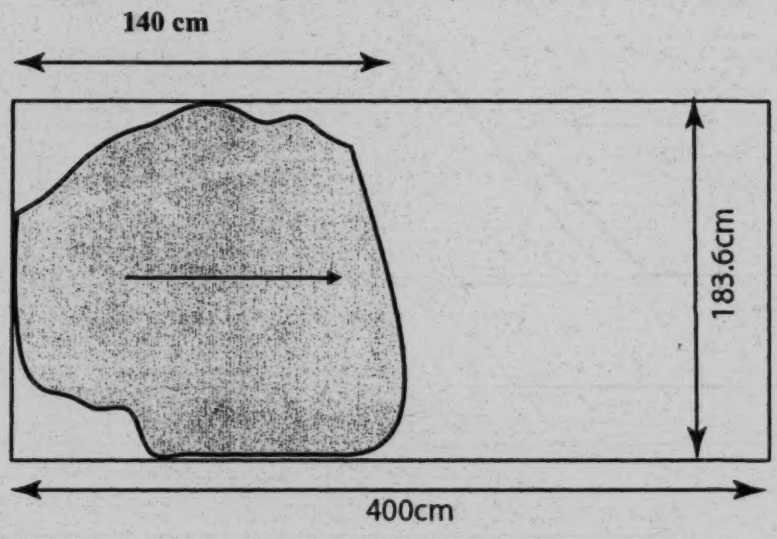

Figure 4-13 Flow profile with salt concentration of 20% salt

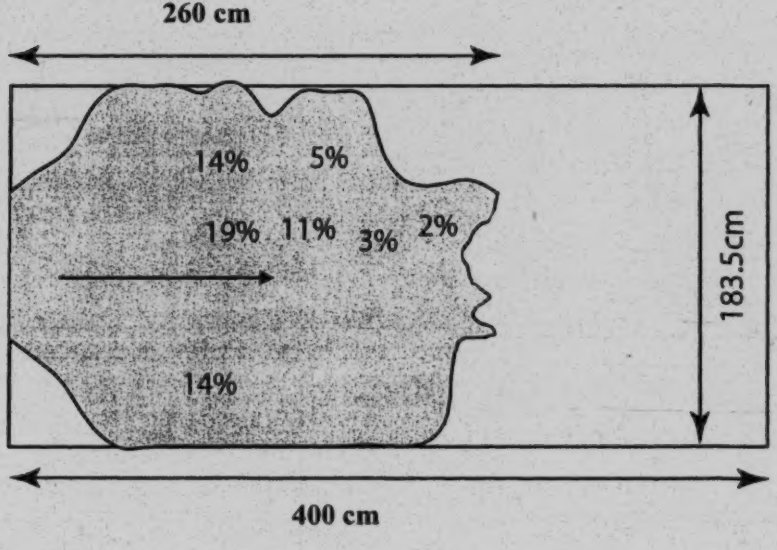

Figure 4-14 Salt concentration at different locations with initial discharge of 4% salt-sulphur mixture (spread over sulphur only layer)

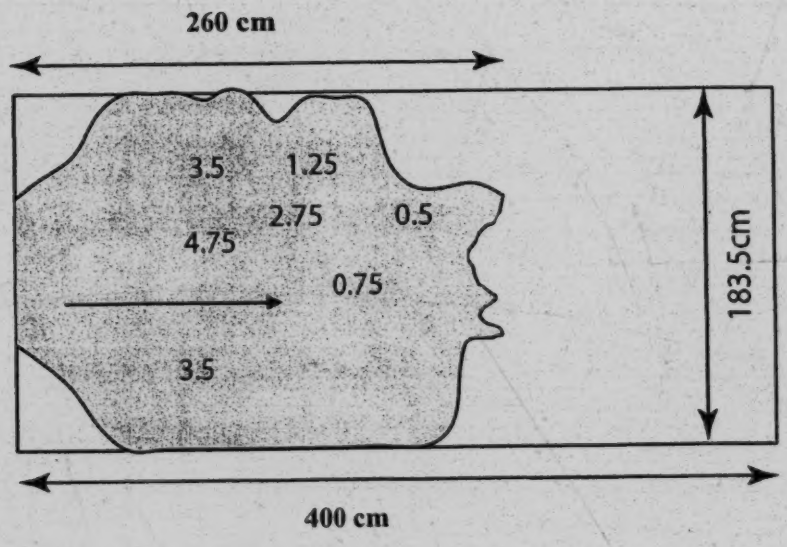

Figure 4-15 Flow diagram normalized to 4% salt concentration (spread over sulphur only layer)

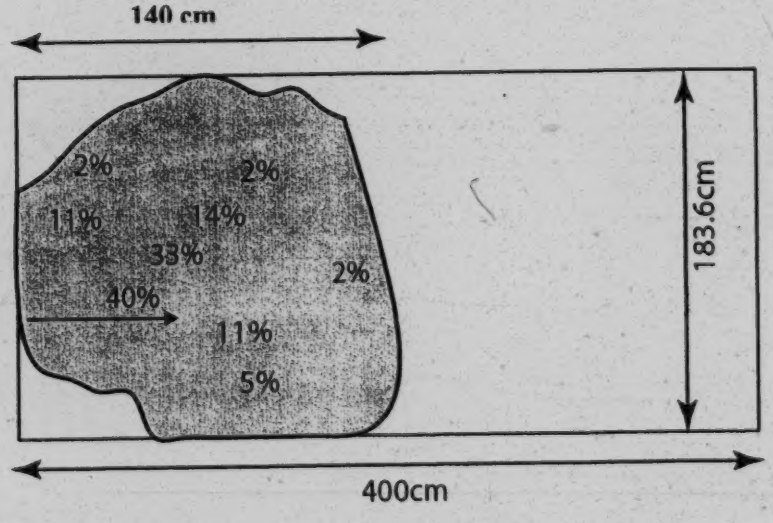

Figure 4-16 Salt concentration at different locations with initial discharge of 20% salt-sulphur mixture (spread over sulphur only layer)

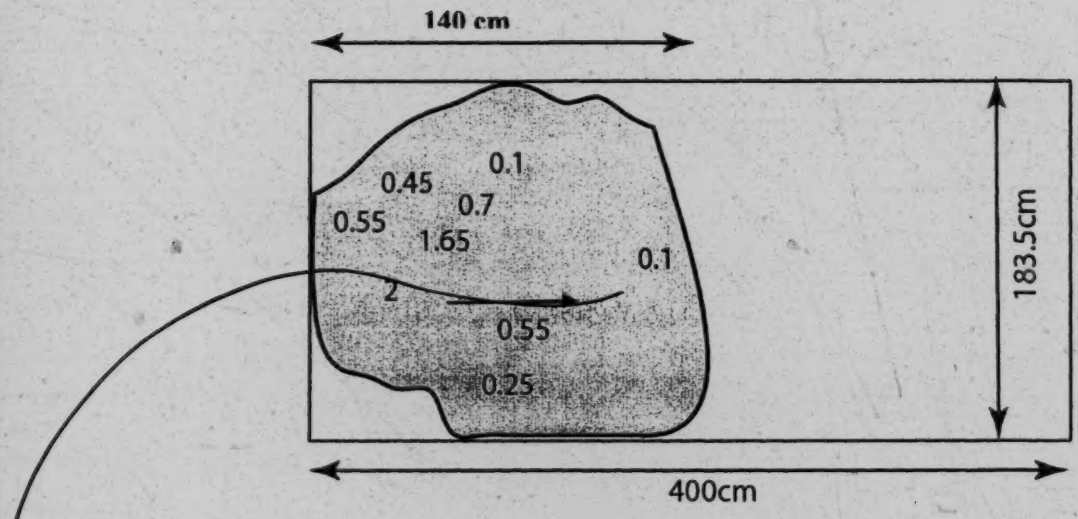

Figure 4-17 Flow diagram normalized to 20% salt concentration

5. DISCUSSION

This chapter discusses the results of the experimental study carried out on elemental sulphur as well as sulphur mixed with salt at different concentrations that were presented in Chapter 4. It also compares the measured results with predictions from the analytical model for similar environmental deposition conditions. The practical implications of this study as they relate to development (design) and operation of a field disposal area conclude this chapter.

The numerical model has limitation and was not developed for analysis of the flow of sulphur salt mixtures. Therefore comparisons were carried out using the physical properties of molten sulphur with the actual sulphur discharge rates. This approach is an approximation to the actual physical flow. The limitation associated with the analytical model relate to measuring the viscosity of the sulphur salt mixtures. A review of the literature and discussion with Drs. Hani Henein and Michael C. Williams in Chemical and Material Engineering department did not result in being able to develop an acceptable laboratory test method to determine the viscosity of this mixture of molten sulphur and salt. As the sulphur salt mixture is a segregating mixture, as it is discharged from the pipe, the viscosity characteristics of the mixture continually change as its flow. This continual change is due to the ongoing settling of the salt particles from the mixture.

5.1 Flow Behavior of Sulphur

Sulphur was discharged at mass flow rates of 13.7 and 14 kg/sec-m in tests 1 and 5, respectively. The significant lengths observed were 3.2 and 3.7 m, respectively. Although the flow rates were almost identical the difference in significant lengths observed was attributed to differences in the outside air / platform temperature during each test. The outside air / platform temperature for test 1 was 4.8 °C and for test 5 it was 15.8 °C.

Figure 4-8 compares the results obtained in tests 1 and 5 with the results from the analytical model. The results from the analytical model are 13 % and 5 % greater than

the significant lengths measured in the field for 4.8 °C and 15 °C respectively. The comparison shows that the difference between the actual and modeled significant length is less when the outside temperature is warmer. The results shown in Figure 4-1 showed that under a constant outside air / platform temperature, an increase in sulphur discharge temperature would increase the significant length at different discharge temperatures. The reason is a longer length is required for the convection and conduction heat losses to cool the mass thus changing it viscosity prior to the sulphur undergoing phase change into a solid. As the temperature of either the sulphur or outside air increases, the significant length increases. Conversely if there are larger differences between the ambient outside air and sulphur discharge temperature, the heat losses will be more rapid hence resulting in shorter significant lengths. The significant length as outlined in Otto (2002) is a function of the rate of heat loss from the sulphur both to cold sulphur below (conduction) and the ambient air above (convection).

The model has limitations and assumptions and uses parameters that have been obtained from the literature (Otto, 2002). Any variation of these parameters will result in a different calculated significant length. Overall there is good agreement between the calculated and measured significant length when using the parameters obtained from the literature for flow of sulphur.

5.2 Sulphur mixed with Salt

Five tests were performed with varying salt concentrations (4%, 9%, 15%, 20% and 25%) in the sulphur. The flow of salt mixed with sulphur at 25 % could not be performed due to a blockage of the discharge pipes. The flow pattern (profile) of salt mixed with sulphur was observed and described after the each test in Chapter 4. The measurements of these flow patterns were taken using tape measurements in the longitudinal direction (significant length) and transverse direction at the test platform. The discharge outlet was considered as the reference discharge point. Samples were collected at random locations to determine the quantity of salt deposited with the sulphur. Figures 4-10 to 4-13 show the flow profiles at different salt concentrations.

The analytical results for sulphur only discharge gave significant lengths within 15 % of the actual test results. Although the numerical model is not valid for salt and sulphur mixed case, a comparative trial run was carried out by taking the equivalent flow discharge based on only the sulphur contained in each mixture. The quantity of salt was excluded from the combined mass flow rate to arrive at this equivalent mass flow rate of sulphur only. This equivalent mass flow rate was then used in the numerical model and a significant length was calculated simulating the predicted sulphur only flow. The significant length measured during the field test for sulphur only flow case, at a particular salt concentration and the significant length obtained from numerical model for the simulated sulphur only case were compared. This comparison of significant lengths was carried out to check if the numerical model can be used for prediction of significant lengths for salt mixed sulphur. Table 5-1 presents the summary of significant lengths prediction at different equivalent sulphur mass with varying salt concentrations. The plots in Figures 5-1 to 5-4 show a comparison of experimental and analytical values of significant length.

Figure 5-1 presents a comparison of the three significant lengths for the sulphur mixed with 4% salt. The significant length measured in the field for sulphur only at a mass flow rate of 14 kg/sec-m at the air temperature of 15 °C is 3.7 meters. Significant length obtained in the field for a 4% salt-sulphur mixture at a mass flow rate of 16.5 kg/sec-m at air temperature of 14 °C is 2.6 meters. The significant length calculated by using numerical model at a mass flow rate of 15.84 kg/sec-m at temperature of 15 °C is 4.73 meters. The mass flow rate was converted from 16.50 to 15.84 kg/sec-m by removing the salt mass in the calculation on 4% conversion basis. The conversion was made by decreasing the mass of sulphur by 4% which accounts for the amount of salt added during the test. The significant length obtained from numerical model for the equivalent sulphur mass was 4.73 m whereas the length measured in the field was 3.7 m. The numerical model predicted a 27% increase in the significant length as compared to the measured experimental significant length.

Figure 5-2 presents a comparison of the three significant lengths for the sulphur mixed with 9% salt. The significant length measured in the field for sulphur only at a mass flow rate of 14 kg/sec-m at the air temperature of 15 °C is 3.7 meters. Significant length obtained in the field for a 9% salt-sulphur mixture at a mass flow rate of 15 kg/sec-m at air temperature of 14 °C is 2.8 meters. The significant length calculated by using numerical model at a mass flow rate of 13.65 kg/sec-m at temperature of 15 °C is 4.07 meters. The mass flow rate was converted from 15 to 13.65 kg/sec-m using the 9% conversion basis. The conversion was made by decreasing the mass of sulphur by 9% which accounts for the amount of salt added during the test. The significant length obtained from numerical model for the equivalent sulphur mass was 4.07 m whereas the length measured in the field was 3.7 m. The numerical model predicted a 9% increase in the significant length as compared to the measured experimental significant length.

Figure 5-3 presents a comparison of the three significant lengths for the sulphur mixed with 15% salt. The significant length measured in the field for sulphur only at a mass flow rate of 14 kg/sec-m at the air temperature of 15 °C is 3.7 meters. Significant length obtained in the field for a 15% salt-sulphur mixture at a mass flow rate of 13 kg/sec-m at air temperature of 18 °C is 2.1 meters. The significant length calculated by using numerical model at a mass flow rate of 11.05 kg/sec-m at temperature of 18 °C is 3.28 meters. The mass flow rate was converted from 13 to 11.05 kg/sec-m using the 15% conversion basis. The conversion was made by decreasing the mass of sulphur by 15% which accounts for the amount of salt added during the test. The significant length obtained from numerical model for the equivalent sulphur mass was 3.28 m whereas the length measured in the field was 3.7 m. The numerical model predicted a 11% decrease in the significant length as compared to the measured experimental significant length.

Figure 5-4 presents a comparison of the three significant lengths for the sulphur mixed with 20% salt. The significant length measured in the field for sulphur only at a mass flow rate of 13.7 kg/sec-m at the air temperature of 4.8 °C is 3.2 meters. Significant length obtained in the field for a 20% salt-sulphur mixture at a mass flow rate of 16 kg/sec-m at air temperature of 8 °C is 1.4 meters. The significant length calculated by

using numerical model at a mass flow rate of 12.8 kg/sec-m at temperature of 5 °C is 3.66 meters. The mass flow rate was converted from 16 to 12.8 kg/sec-m using the 20% conversion basis. The conversion was made by decreasing the mass of sulphur by 20% which accounts for the amount of salt added during the test. The significant length obtained from numerical model for the equivalent sulphur mass was 3.66 m whereas the length measured in the field was 3.2 m. The numerical model predicted a 14% increase in the significant length as compared to the measured experimental significant length.

Figure 5-5 shows the comparison of measured significant lengths at different salt concentrations with the sulphur. It is observed that as the salt concentration increases the significant length decreases. The decrease in significant length is more prominent when the salt percentage is 15% and 20%. This decrease in significant length is due to the transverse flow of sulphur resulting from hydraulic sorting of salt particles.

The concentration of salt in sulphur ranged from 4 % to 20 % by mass. The salt concentration limits were chosen such that the flow behavior of sulphur will not change drastically and that sulphur would act as the carrier fluid for the salt during these tests. Similar mixtures were also used by Ding (2006) in his study on the environmental impacts of salt and sulphur disposal in the mined out pit.

The sulphur mixed with salt produce a segregating mixture that deposited the higher concentrations of salt near the discharge outlet and then dispersed in a transverse and longitudinal direction with decreased salt concentrations at the limits of flow region. The salt concentration near the flow outlet dictates the flow behavior of the sulphur and salt mixture. It was observed that although the general flow direction of the salt and sulphur mix was in the longitudinal direction away from the outlet and along the platform, some flow occurred in the transverse direction. The flow behavior can be termed braided in nature. The transverse flow was not significant when pure elemental sulphur alone was discharge. It was observed that the significant length decreases as the concentration of salt increases. With increasing salt concentrations the transverse flow increased in significance. Figures 4-10 to 4-13 show the individual flow patterns of each

mixture of sulphur and salt. These flow patterns confirm that concentration of coarser salt particles occurs near the discharge outlet. The concentration of finer particles was observed further along the discharge surface. This phenomenon is termed hydraulic sorting (refer to Section 2.3), and is shown in more detail in Figure 4-14 and 4-16.

Figure 5-6 shows a comparison of significant length obtained for the sulphur experiment to the significant lengths for varying salt concentrations (4%, 9%, 15% and 20 %) using the equivalent sulphur mass flow rate. This comparison was carried out to check the viability of using the model for prediction of the flow behavior of sulphur and salt mixture in the field. The limitation of model is that it only encompasses the properties of sulphur and does not account for the properties of any material mixed in sulphur. This comparison was carried out using the equivalent sulphur discharge rates (the mass flow rate reduce for the proportional weight of salt added). This was achieved by subtracting the mass of salt from the sulphur and salt mixture and using only the mass of sulphur in the calculations. It is observed in Figure 5-6 that the significant lengths evaluated using the analytical model for equivalent sulphur discharge rates remain within about +/- 15 % deviation from the significant length measured in the field test for sulphur alone.

The behavior of salt mixed with sulphur is identical to the behavior of segregating slurry of sand and water as observed by Küpper (1991) in her thesis on the design of hydraulic fill. Küpper outlines that the behavior of hydraulically placed tailings depends primarily on its fabric and grain size distribution, which in turn is dependent on how the material was deposited. The segregating slurry deposits the coarser fraction of solids closer to the discharge point and the finer fraction farther away.

Hydraulic sorting refers to the process of deposition of particles of different sizes at different distances from the discharge point. Larger particles tend to deposit soon after being discharged, while small particles can be carried by the flow and deposited further downslope. Hydraulic sorting is more pronounced for high discharge rates, small slurry concentrations and relatively low flow velocity. The Soviet standard specification SNiP-

11-53-73 recommends consideration of sorting if the material has $D_{60}/D_{10} > 2.5$ and/or $D_{90}/D_{10} > 5$; although hydraulic sorting can start for $D_{60}/D_{10} \sim 1.3$ to 1.6. The D_{60}/D_{10} for the salt used in the field tests is 5.7 and D_{90}/D_{10} is 22. These values clearly indicate a possibility for hydraulic sorting to occur in the salt and sulphur mixture when it is discharged in a sulphur blocking facility.

Typical flow of segregating sand and water slurry is a complex combination of sheet flow, braided flow and meandering channels with associated formation of bars, islands and lag deposits. Similar conditions are expected for a slurry composed of salt within the sulphur. Sheet flow commonly occurs right near the discharge point, where the flow spreads and the specific flow rate is relatively high. After the flow opens up significantly the specific flow rate decreases and the material compensates by settling to form braids, islands and bars. This is the behavior that was observed in this study and is illustrated in Figures 5-7 to 5-8.

The trend in Table 4-5 shows that as the salt concentration is increased the flow is dispersed over the platform and hence a higher percentage of significant length is covered in transverse direction. With the 4% salt concentration the hindrance of flow due to salt was minimal and sulphur covered all the area of platform. But as the salt concentration increases more area of platform in the transverse direction is covered. In case of 9% salt concentration 100% of initial one third of platform width is covered followed by 30% each in next two third portions. In case of 15% salt concentration initial 100% is covered followed by 70% and then 30% in next third portion. Now in case of 20% salt concentration all of the significant length is covered across the platform. This showed as the salt concentration increases, hydraulic sorting becomes more dominant resulting in significant flow in transverse direction.

5.3 Implications of Results for Field Operations

The working temperature range of sulphur is limited to between 115 to 160 °C. This is the temperature range where sulphur is a fluid with its minimum viscosity and is

the only practical temperature range where sulphur can be a carrier fluid. Any temperatures outside this range will have a pronounced adverse effect on the flow properties due to the viscosity changes in the sulphur.

A broad gradation of salt for this test analysis has been used to find out the flow behavior of salt mixed with sulphur for various particle sizes (coarse to fine particulate). This gradation has also been used by Ding (2006) in the parallel study. Based on the findings of this study, it is recommended that if fine grained salt particles (clay size) are to be deposited the mixture might behave as non-segregating slurry producing a more uniform flow pattern and a homogeneous disposal solid mass. The phenomenon of non-segregating slurry behavior is also discussed in detail by Küpper (1991).

The platform configuration used for this study was limited in dimensions compared to the actual mined out pit that may be used as a disposal area. Dimensions of a sulphur block in the field as proposed by HAZCO for sulphur storage are about 600 m in length and 600 m in width (Figure 2-6). To extrapolate the significant lengths obtained in the case of sulphur, a calculation was carried out by inputting into the model a realistic field discharge rate for sulphur. The discharge was varied from 1500 to 3000 Tonnes per day (~ 494 to 990 kg/ sec–m). The outside ambient air temperature was assumed at 10 °C. The significant lengths calculated ranged from 100 m to 200 m respectively. These significant lengths serve as an estimate for determining the dimensions of sulphur block depending on the deposition method adopted for either sulphur or salt mixed with sulphur (section 2.1.3).

For the case of salt mixed with sulphur, the flow pattern indicated that substantial flow in the transverse direction compared to the longitudinal direction occurred for salt concentrations greater than about 10 %. The significant lengths for salt mixed with sulphur having concentration greater than 10 % will be reduced to 50 to 100 m in the longitudinal direction. This is due to the substantial amount of flow in the transverse

direction resulting from the flow being redirected due to segregation and settling of the salt from the sulphur following discharge.

The flow direction using the analytical model in this study has been uni-directional while the experiments were carried out using discharge from a single pipe. The maximum distance the material flowed in the experiment is similar to the significant length calculated using the analytical model. The flow pattern may change as a result of multiple outlets to discharge on a surface or plane. A single point discharge with the pipe directed vertically downward will result in radial flow during which the material spreads causing the circumference of the deposit to increase. The maximum radius will be less than the significant length from the analytical mode because the specific discharge will spread over a much larger area.

Based on this study it is observed that the flow behavior of salt mixed with sulphur is similar to that of elemental sulphur, the main reason being that the flow properties of sulphur dominates even when salt is mixed in with sulphur at the low concentration studied. This behavior is expected to hold for up to about 10 % salt within the sulphur by mass. At concentration of salt greater than this may result in a flow behavior that is more dominated by hydraulic sorting of the salt from the mixture.

The model available for determination of significant lengths has only been confirmed for the flow of liquid sulphur. In the case where salt is mixed with sulphur the significant length needed for design purposes requires the use of the equivalent sulphur mass flow rates in the model to predict the significant lengths. The predicted significant lengths are within +/- 15 % of the significant length measured for the different salt mixtures tested during this research.

5.3.1 Design Recommendations for a Sulphur Block

The daily production of sulphur at Syncrude is about 2700 tonnes per day. In addition, 400 tonnes of salt is also expected to be produced. The safe disposal

methodology for this salt and sulphur mass was the main focus of this research study. The mass ratio of salt to sulphur for this scenario is 15 % salt to sulphur mass.

For design purposes an initial discharge temperature of 120 °C with an outside air temperature of 23 °C (maximum temperature at Fort McMurray in the year 2003, maximum temperature produces maximum significant lengths) are considered. The discharge of salt mixed with sulphur when using a 25 mm (1 inch) diameter pipe and only one stationary discharge outlet is 890 kg/sec-m. Using this mass flow rate the model predicts a significant length equal to 300 m. This significant length is for sulphur alone. If the salt is mixed with sulphur, the predicted significant length is expected to be 15 % less than this analytical predicted length. This reduction in significant length with the addition of salt is attributed to the segregating behavior of the flow associated with these mixtures.

Practically speaking; such high mass flow rates are not possible for a pipe having outlet diameter of 25 mm diameter. One approach to lower the mass flow rate is to increase the number of discharge outlets. This is known as a multiple discharge system or spigot system. The mass flow rate will be distributed over the number of outlets in operation. Considering a 75 mm diameter (3 inch) pipe the possible mass flow rates it can handle is about 300 kg/sec-m. The discharge thus is distributed on each outlet in operation. For a square deposition to create a block, if four outlets discharge sulphur and salt mixture in all four perpendicular directions, the discharge of sulphur alone will be around 250 kg/sec-m. This reduction in mass flow rate will eventually reduce the significant length which is predicted to be about 82 m from each outlet. For outlets opposite one another total significant length becomes 164 m. With the addition of salt, due to the deposition of coarser grains near the discharge outlet and braided nature of flow (flow also occurring in the lateral direction) the 15% compensation will enable the total physical dimension of the block to be reduced to 140 m. It is observed in Fig 4-14 and 4-16 that the maximum salt concentration is in the initial one third portion of sulphur and salt mixture flow. In regards to this observation the maximum salt deposition in the block is expected to be within the first one third of the significant length due to hydraulic

sorting i.e. 25 m from each discharge point. A block with a foot print area of 150 x 150 m^2 is therefore recommended for disposal. The central portion of the block will however will contain less coarser salt fraction and will only contain the finer fraction. Salt will therefore be concentrated at the edges of the blocks. To have uniform salt concentration within the block the discharge outlets will need to be moved.

Considering the daily flow volume as 3100 tonnes / day (sulphur and salt combined) and the density of sulphur to be 1800 kg/m^3. The volume to be disposed of is 1722 m^3 per day. Considering a foot print area of 150 x 150 m^2 (22500 m^2), a lift thickness per day is 7.75 cm. Considering a limiting height of 12 m (AMEC, 2003), the block is expected to reach a height of 12 m in 150 days. The total sulphur and salt storage capacity per block will be 258,300 m^3.

An alternative approach is to follow the recommendations given by AMEC 2004 (section 2.1.3.1.1) and design a block that disposes of 500 m^3 /day of sulphur and salt. The mass flow rate would be 100 kg/sec-m considering a 7.5 cm (3 inch) diameter pipe. Reducing the mass flow rate for the 15 % salt concentration, the equivalent sulphur mass flow rates will be 85 kg/sec-m. The significant length obtained from the model is only 28 m. With the addition of salt, due to the deposition of coarser grains near the discharge outlet and braided nature of flow (flow also occurring in the lateral direction) the 15% compensation will enable the total physical dimension of the block to be reduced to 25 m. For a rectangle block formation, if moving the outlet is an option used then the footprint area is 50 m x 200 m (Figure 5-10). The maximum salt concentration due to hydraulic sorting is expected to be within 8 m of the outer perimeter of the block. The central portion of the block will however contain less of the coarser fraction and will only contain the finer fraction.

If the discharge outlet is moved, it is recommended to discharge sulphur and salt mixture in sections within the block. Hence the lift thickness for pouring 500 m^3 daily is 5 cm. Considering a limiting height of 12 m (AMEC, 2003), the block is expected to

reach a height of 12 m in 240 days. The total sulphur and salt storage capacity will be around only 120,000 m^3.

The above discussion shows that due to hydraulic sorting of the non-segregating slurry, 75 % of the salt concentration remains around the perimeter of the blocks. To overcome this situation and to capture the maximum salt content within the central portion of block it is recommended to discharge the flow in the central region of the block so that flow occurs towards the outer edges. This will form a cell with maximum salt concentration near the center (Figure 5-11). This will form a circular block. If this flow is not confined with the form work on the sides the shape of circular block would be in the form of cone at the top (Figure 5-12).

Considering a circular block that can contain 2700 tonnes sulphur and 400 tons salt production per day, the total discharge to handle this daily amount is 890 kg/sec-m. With a 7.5 cm diameter pipe and four outlets for discharge, the individual discharge capacity would be 250 kg/sec-m for each pipe. The expected significant length from each outlet would remain 82 m. Allowing for the reduction in significant length associated with the mixture results in a salt mixed with sulphur the significant length of 70 m. A circular block with perimeter fabricated of straight panels (form work) is recommended. The radius of 70 m will give a foot print area of 15,394 m^2. The maximum concentration of salt would be within one third the distance (23 m) from the central discharge outlets (Figure 5-11).

Considering the daily flow volume as 3100 tonnes / day (sulphur and salt combined) and the density of sulphur to be 1800 kg/m^3. The volume to be disposed of comes out at 1722 m^3 per day. Considering a foot print area of 15000 m^2 (70 m radius), a lift thickness for pouring of this daily discharge comes out to be 11 cm. Considering a limiting height of 12 m (AMEC, 2003), the block is expected to reach a height of 12 m in 110 days. The total sulphur and salt storage capacity will be around 180,000 m^3.

Ding (2006) reports that the least environmental impacts for salt disposal in sulphur occur with the salt being encapsulated in the central region of a sulphur block. Figure 5-11 shows the preferred location of salt body within the sulphur block.

Internal crack in the sulphur blocks at Syncrude have been observed. Packer test used to measure the hydraulic conductivity of sulphur blocks gave values in the order of 10^{-1} m/sec (Syncrude, 2004). This extremely high hydraulic conductivity of sulphur blocks result in rain water passing into and through the block.

Environmental protection services of Alberta Environment, water quality standards and approvals and pollution control division undertook a survey of a representative number of sulphur plants across Alberta to assess the adequacy of the present run-off controls in which it was observed that pH of run-off water was very low, pH 1 to 2 in some case (Alberta Environment, 1978). This causes many environmental concerns like contamination of ground water and destruction of adjacent vegetation. Environmental protection services in their report on surface runoff from sulphur storage blocks in Alberta (AENV, 1978) recommended improving overall housekeeping of the sulphur block.

One reason for this cracking is the shrinkage behavior of sulphur as it solidifies and cools. Liquid sulphur at 120 °C has density of 1803 kg/m^3 and solid sulphur has a density of 1920 kg/m^3. This increase of 6% in its density results in a noticeable shrinkage of sulphur as it cools and solidifies.

Now considering the design of the sulphur block proposed for a discharge of 3100 tonns / day, the volume disposed of comes out to be 1722 m^3 / day. Considering 6% shrinkage means that 103 m^3 of shrinkage occurs each day as the sulphur solidifizes. This shrinkage during phase change results in cracks forming within the block. To overcome this shrinkage it is proposed to provide shrinkage joints at predetermined location so that any shrinkage that occurs takes place at these joins instead of producing cracks within the sulphur being placed. In case of a circular block with radius of 70 m

and lift thickness of 11 cm per day it is proposed to provide a radial shrinkage joint at a spacing of 30° from each other, this means 12 radial joints for this circular block. As the maximum concentration of salt is within the initial one third of the significant length from the discharge outlet, one circumferential joint is proposed at distance of 25 meters from the discharge (Figure 5-13). These joints could be filled with Bentonite or a geocomposites containing Bentonite. As the liquid sulphur solidifies it shrinks, away from these Bentonite filled joints and a gap is formed. The next day the sulphur will flow over into this gap thus filling the gaps formed due to previous day's shrinkage and then the sulphur will spread. This process will be repeated for all successive pourings and a sulphur block with a minimum of cracks can be achieved. M/s CETCO linings technologies produces Bentonit ST or Bentonit SDN which can be installed to form the shrinkage joints. These have not been tested at a high temperature of 120 °C. However a custom design can be requested to CETCO or any other manufacturer for high temperature geotextile to act as a container with high shear strength to allow the product to be self supporting so it can form these joints.

The tensile strength of sulphur need to be examined for the proposed block dimensions to ensure that the formation of these cracks is not due to tensile failure. The proposed design based on shrinkage is consider being conservative and will result in construction of a block that will minimize random cracks within the blocks.

5.4 Summary of Design Recommendations

Different options for the design of sulphur storage blocks are recommended in section 5.3 and summarized in this section.

1. Considering the flow of 2700 tonnes/day for sulphur and 400 tons per day of salt a 150 x 150 m^2 block is recommended with discharge outlets at the outer perimeter. The lift thickness in this case will be 8 cm and the block will reach a height of 12m in 150 days. This block will facilitate the 258,000 m^3 of sulphur.

2. Considering the flow of 500m^3 per day of sulphur and salt a block of size 50m x 200m is recommended. This block with the lift of 5 cm will reach the height of 12m in 240 days and will accommodate 120,000 m^3 volumes of sulphur and salt.

3. Circular sulphur block of radius 70m is recommended for the 2700 tons/day of sulphur and 400 tons/day of salt. With the lift thickness of 11 cm this block will attain the height of 12m in 110 days and will accommodate the volume of 180,000 m^3. This circular block will have central discharge system and considered as most feasible for sequestering the salt in sulphur.

4. In order to avoid cracks in the sulphur blocks it is recommended to provide shrinkage joints at predetermined location at the spacing of 30° across the circular block. These joints could be filled with bentonite or a geocomposite containing bentonite.

	Mass rate of flow kg/sec-m	T_{air} °C	Significant length (m)
Sulphur only	14	15	3.7
Sulphur only	13.7	4.8	3.2
Sulphur + 4% salt	16.5	14	2.6
Sulphur + 9% salt	15	14	2.8
Sulphur + 15% salt	13	18	2.1
Sulphur + 20% salt	16	8	1.4
Equivalent sulphur mass @ 4%	15.84	15	4.73
Equivalent sulphur mass @ 9%	13.65	15	4.07
Equivalent sulphur mass @ 15%	11.05	18	3.28
Equivalent sulphur mass @ 20%	12.8	5	3.66

Table 5-1 Prediction of significant lengths at different equivalent sulphur mass

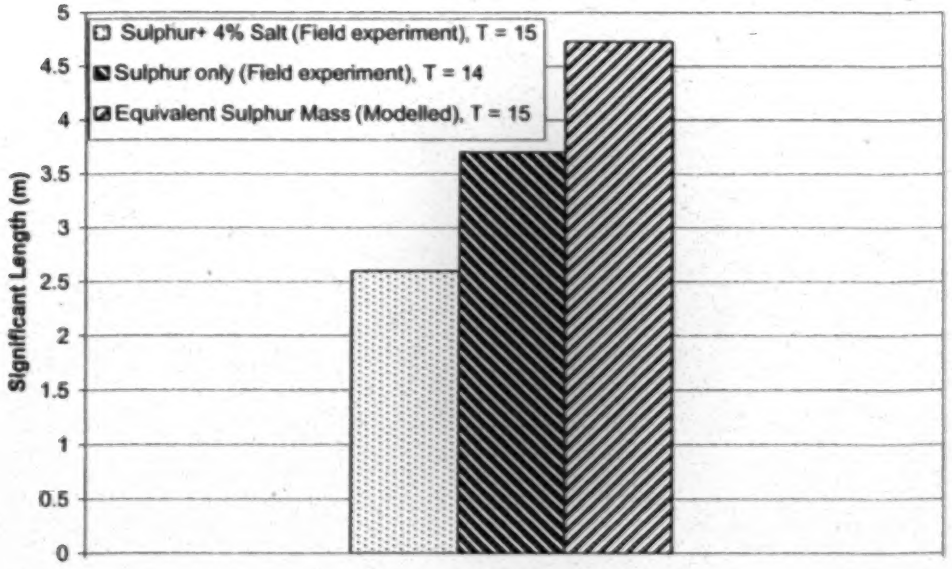

Figure 5-1 Prediction of significant length for equivalent sulphur mass 4% salt concentration

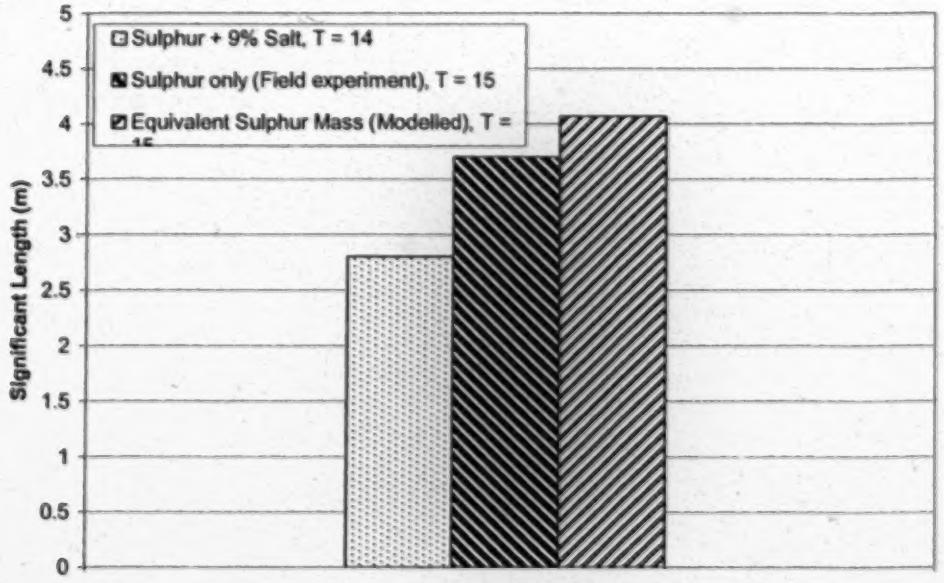

Figure 5-2 Prediction of significant length for equivalent sulphur mass 9% salt concentration

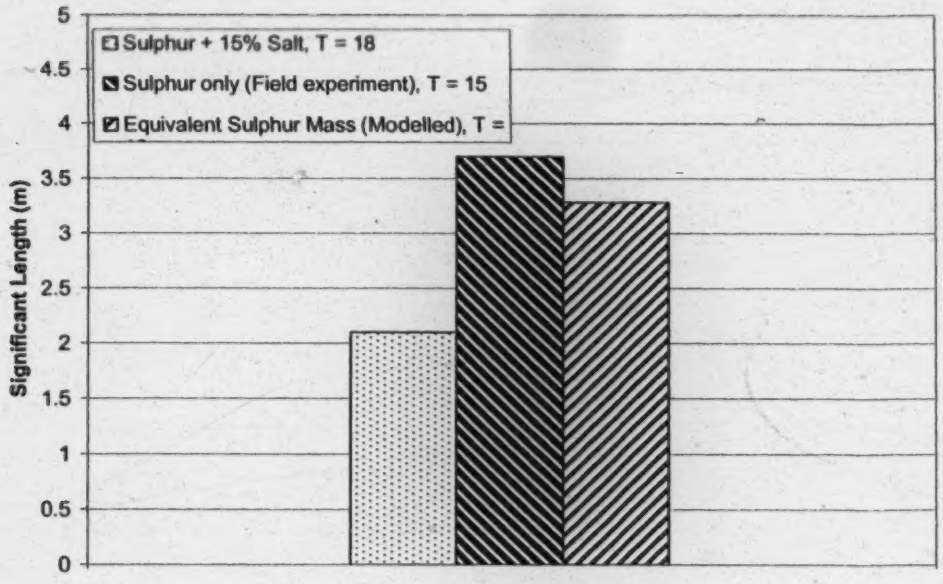

Figure 5-3 Prediction of significant length for equivalent sulphur mass 15% salt concentration

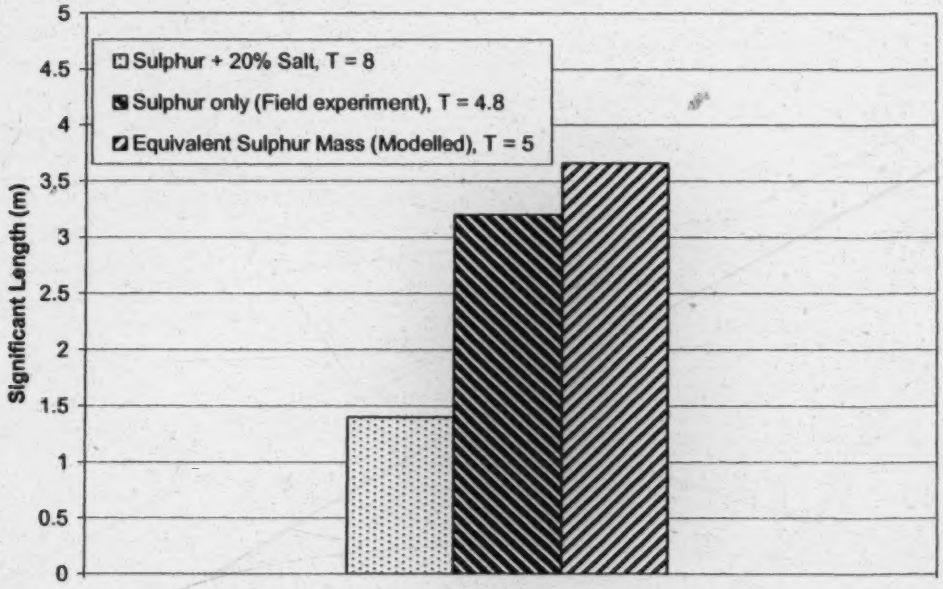

Figure 5-4 Prediction of significant length for equivalent sulphur mass 20% salt concentration

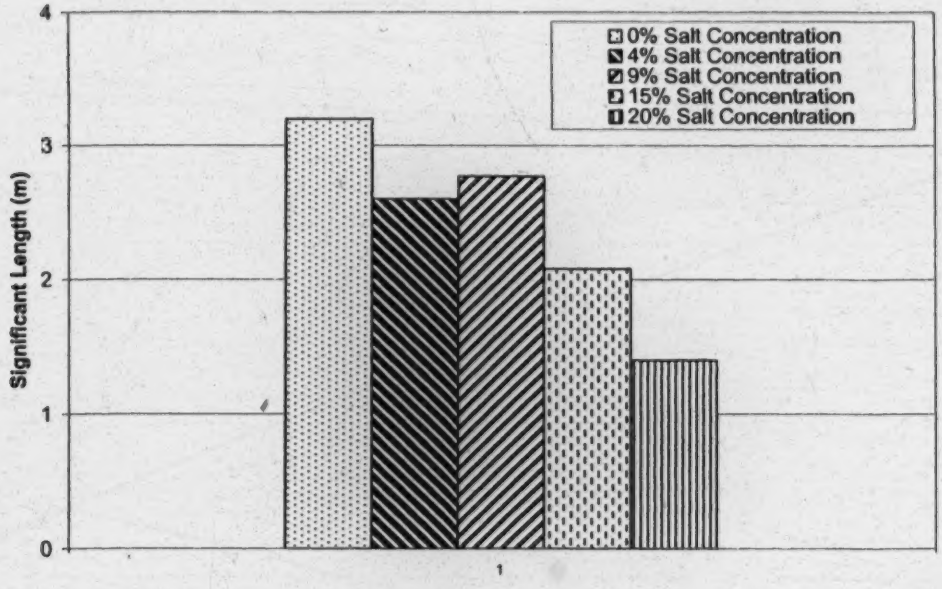

Figure 5-5 Significant length at varying salt concentration

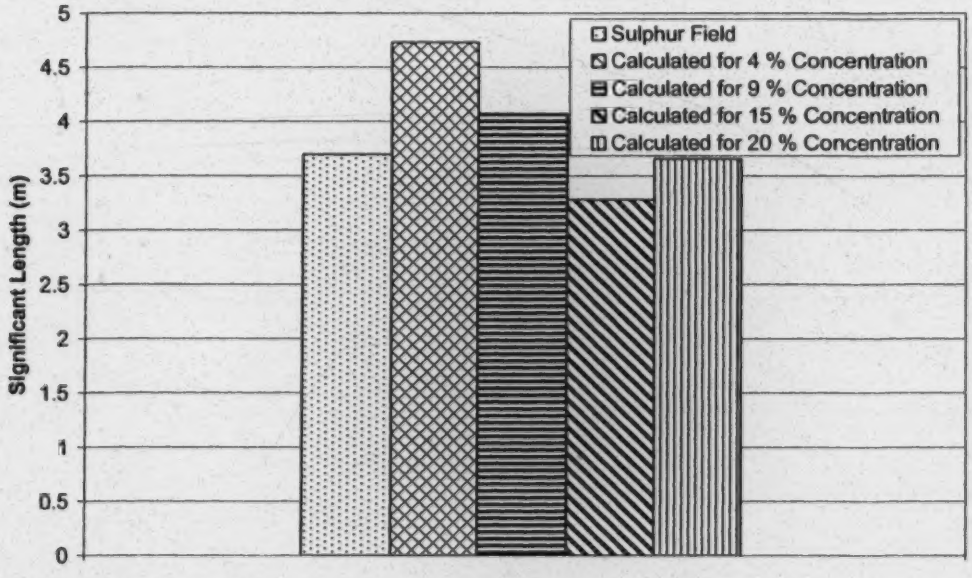

Figure 5-6 Prediction of significant lengths for equivalent sulphur mass

Figure 5-7 Photograph showing hydraulic sorting phenomenon.

Figure 5-8 Photograph showing formation of islands and bars at the flow boundaries.

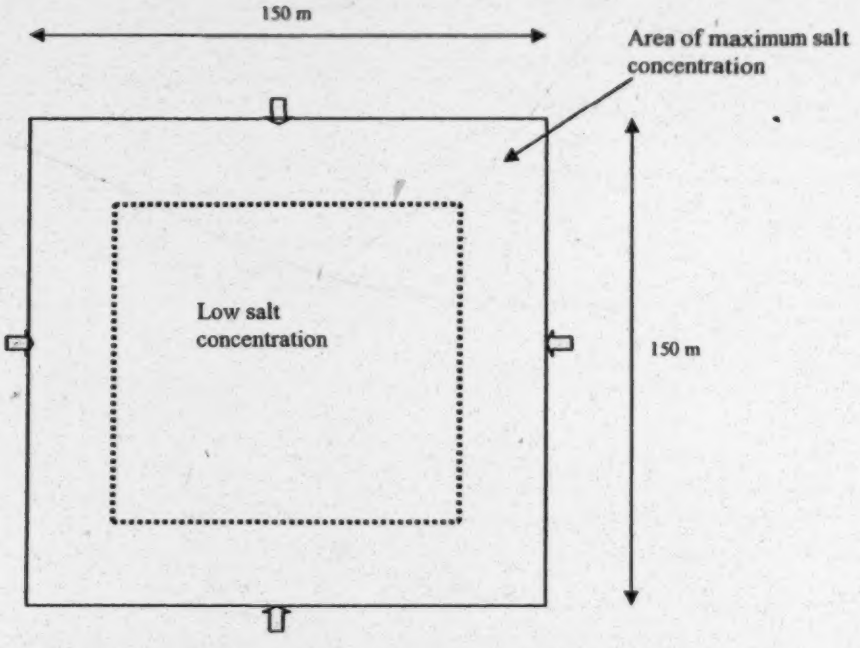

Figure 5-9 General layout of sulphur and salt mixed storage block with side discharge outlets.

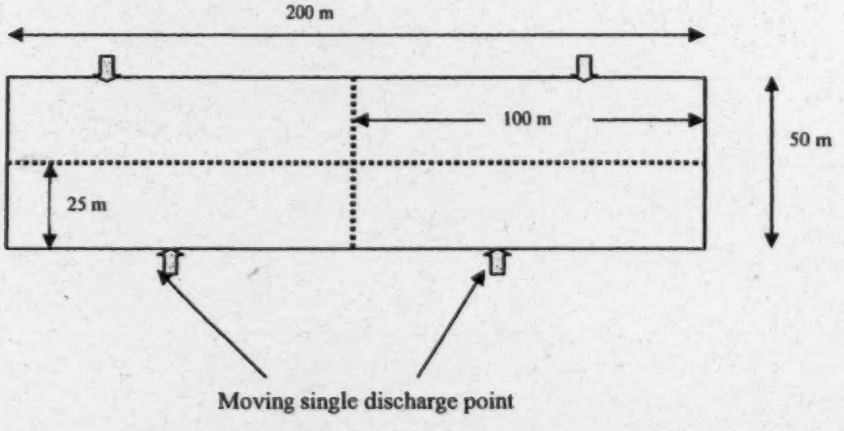

Figure 5-10 General Layout of Sulphur and salt mixed storage block with side mobile outlets

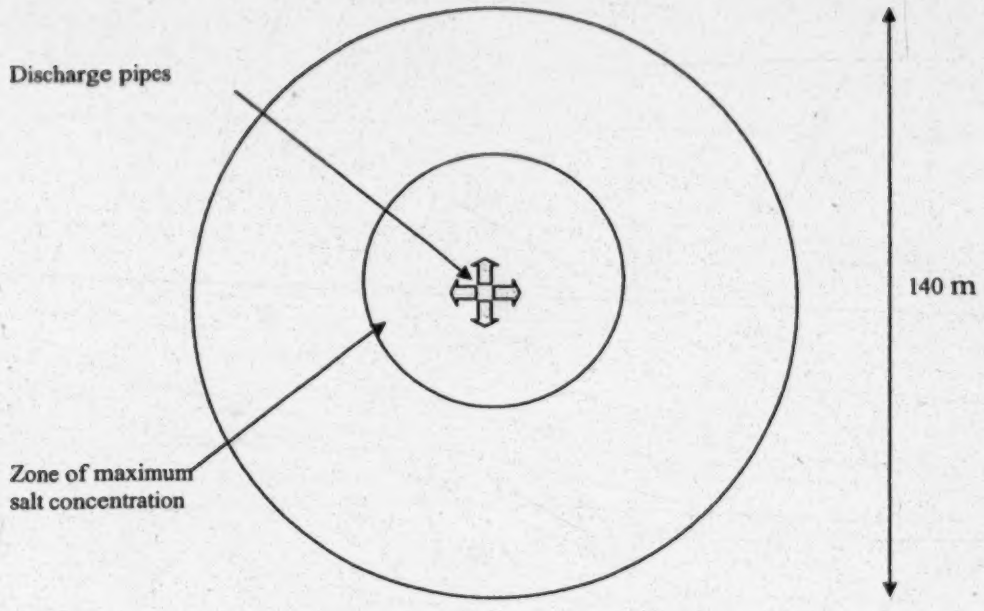

Figure 5-11 General Layout of Sulphur and salt mixed circular storage block

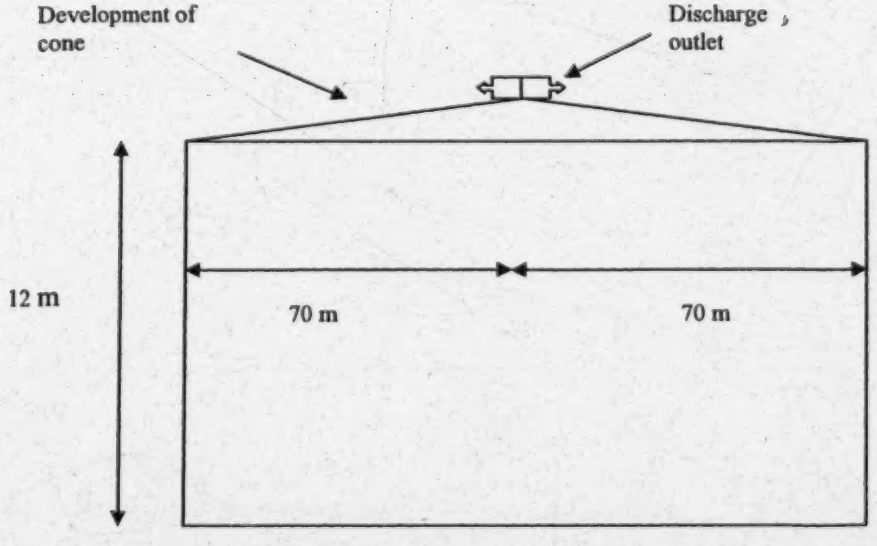

Figure 5-12 Formation of cone with central discharge system in circular block

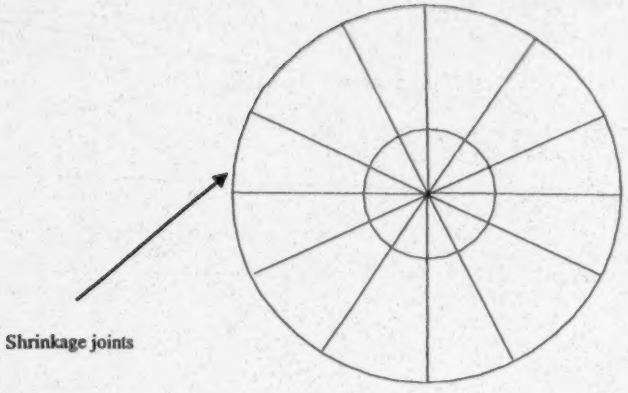

Figure 5-13 Plan view of sulphur block with shrinkage joints

6. SUMMARY AND CONCLUSIONS

6.1 Summary

An elaborate test program as well as an analytical model simulation was carried out to evaluate the feasibility of permanently sequestering of the salt crystal within solid elemental sulphur. Various technical aspects of disposing of the molten sulphur mixed with salt within the mined out pit at Syncrude mine site were evaluated.

Specific references about the flow behavior of sulphur were not available in the literature. Hence special apparatus and equipment was fabricated to meet the required objectives of the test program. This included but was not limited to the dimensions of platform to capture flow behavior of molten sulphur mixed with salt, capsule to contain molten sulphur at the required temperature with an oil bath provision to safely heat the sulphur.

Seven tests were performed using different salt concentrations and the surface discharge profiles were observed. The first test was performed using only sulphur, and the fifth test was performed to verify the results of this first test. The remaining four tests were performed with varying concentration of salt added to sulphur. The tests performed using sulphur were then compared to the model presented and described by Otto (2002).

The results show that if the temperature of either the sulphur or outside air increases, the significant flow length of the sulphur increases. Salt mixed with sulphur was a segregating mixture which shows the concentration of deposited salt to be greater near the discharge outlet and the concentration dispersed at the limits of flow pattern. Hydraulic sorting phenomenon was observed in the flow behavior of salt mixed with sulphur. The salt concentration near the flow outlet dictates the flow behavior of sulphur and salt mixture. It was observed that although the general flow direction of salt and sulphur mix was in the longitudinal direction along the platform, some flow diverted in the transverse direction. It was observed that the significant length decreases as the concentration of salt increases.

Other observations include that maximum salt concentration due to hydraulic sorting is near the discharge outlet i.e. within one third of the significant length. Hence if flow occurs from the centre of the block outwards, the salt is expected to be encapsulated within the central portion which is a more environmental friendly option (see Ding, 2006).

6.2 Conclusions

The experiments verify that the physical flow of sulphur and salt mixture is governed by the concentration of salt in the mixture, and on either the ambient or sulphur temperature. Maximum salt concentration due to hydraulic sorting occurs within the initial one third of significant length. This observation is used to assist with the design of sulphur blocks for encapsulating the salt.

Based on the observations of this study, practical guidelines are provided for the design of sulphur blocks at the mine pit site. It is observed that the cracking of sulphur blocks is due to the shrinkage of sulphur during phase change. Guidelines to overcome this shrinkage cracking are proposed in Section 5.5.

6.3 Recommendation for Future Work

Finally, recommendations for further research follow from the current research. The following areas are recommended for further research:

- Development of a larger platform to verify the results obtained from this study under every increasing mass flow rates
- Bi-directional flow should be carried out
- Broadening the gradation of salt mixed with sulphur
- A pilot scale test to be carried out at the Syncrude mine site for real time comparison of results with this study

Nevertheless, the results obtained in this study provide a practical approach towards developing an approximate size of the block for disposal of sulphur and salt at Syncrude mine pit site.

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